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Tank Characterization Report for Single-Shell Tank 241-B-111

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U.S. Department of Energy Contract DE-AC06-87RL10930

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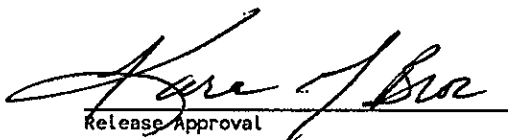B&R Code: EW 3120074   Total Pages: 96

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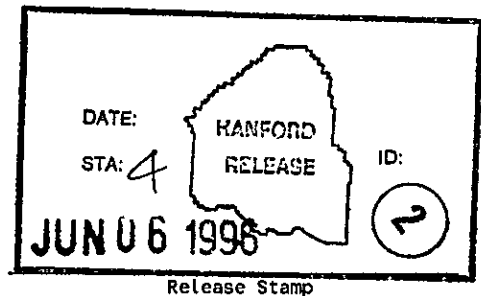
Abstract: This tank characterization report for Tank 241-B-111 was initially released as PNL-10099. This document is now being released as WHC-SD-WM-ER-549 in order to accommodate internet publishing.

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Tank Characterization Report for Single-Shell Tank 241-B-111

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EXECUTIVE SUMMARY

Tank 241-B-111 (hereafter referred to as B-111) is a 2,006,300 L (530,000 gal) single-shell waste tank located in the 200 East B Tank Farm at Hanford. Two cores were taken from this tank in 1991 and analysis of the cores was conducted by Pacific Northwest National Laboratory's 325-A Laboratory in 1993. Characterization of the waste in this tank is being done to support *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) Milestone M-44-05 (Ecology et al. 1994).

Tank B-111 was constructed in 1943 and put into service in 1945 (Table ES-1); it is the second tank in a cascade system with tanks B-110 and B-112. During its process history, B-111 received mostly second-decontamination-cycle waste and fission products waste via the cascade from tank B-110. This tank was retired from service in 1976, and in 1978 the tank was assumed to have leaked 30,300 L (8,000 gal) (Hanlon 1993). The tank was interim stabilized and interim isolated in 1985. The tank presently contains approximately 893,400 L (236,000 gal) of sludge-like waste and approximately 3,800 L (1,000 gal) of supernate. Historically, there are no unreviewed safety issues associated with this tank and none were revealed after reviewing the data from the latest core sampling event in 1991.

Core 29 was taken from riser 3 and core 30 was taken from riser 5 (Figure ES-1). The core recoveries were good (100%), with the exception of segments 2 and 5 from core 30. Since one core was near the waste inlet (core 29) and the other core was taken near the overflow (core 30), these two cores should represent the extreme range of compositions in the tank.

Table ES-1. Engineering Data Summary of Tank 241-B-111.

Tank Engineering Description	
Type:	Single-Shell Tank
Construction:	1943-1944
In-Service:	December 1945
Out of Service:	April 1976
Diameter:	23 m (75 ft)
Operating Depth:	5.2 m (17 ft)
Nominal Capacity:	2,006,300 L (530,000 gal)
Bottom Shape:	Dish
Hanford Coordinates:	N45337.5, W52852.5
Ventilation:	Passive
Tank Status	
Watch List:	None
Interim Stabilized:	June 1985
Interim Isolated:	October 1985
Contents:	Non-Complex Waste
Integrity Category:	Assumed Leaker (1978) (30,300 L [8,000 gal])

An extensive set of analytical measurements was performed on the core composites. The major constituents (>0.5 wt%) measured in the waste are water, sodium, nitrate, phosphate, nitrite, bismuth, iron, sulfate and silicon, ordered from largest concentration to the smallest. The concentrations and inventories of these and other constituents are given in Table ES-2.

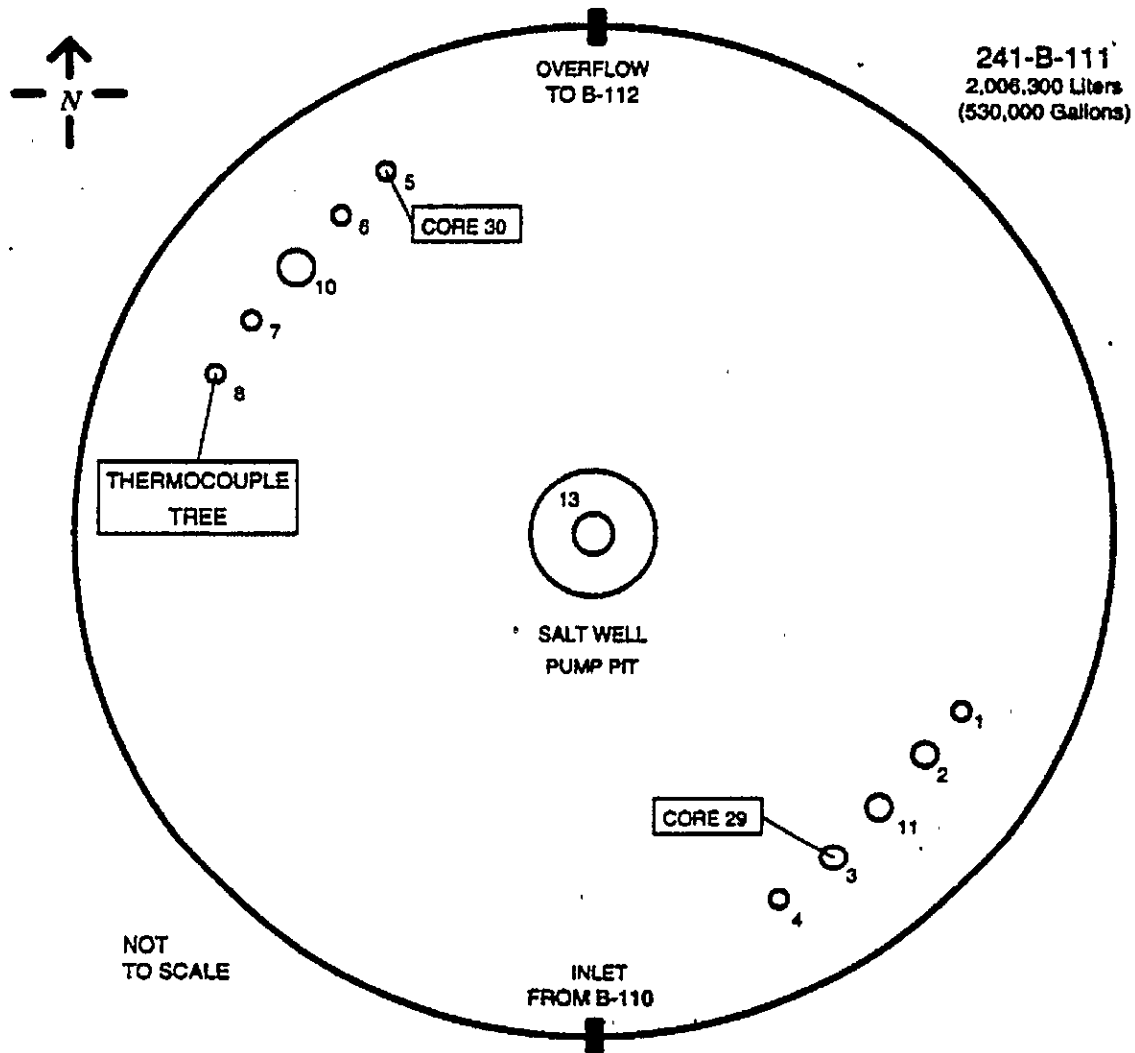Figure ES-1. Top View of Tank 241-B-111.

Table ES-2. Inventory Summary for Tank 241-B-111.

Physical Properties of Waste			
Total Waste:	897,100 L (237,000 gal)	Supernate Volume:	3,800 L (1,000 gal)
Drainable Inter. Liquid:	79,500 L (21,000 gal)	Density:	1.190 g/mL
H_2O Average:	63.1%	Total Waste Mass:	1,067,600 kg
pH:	8.87	Temperature Average:	26.7 °C (80.2 °F)
Heat Load:	2.57e+03 watts	Maximum Exotherm:	No Exotherms
Chemical Properties of Waste			
Sodium:	1.02e+05 kg (9.57 wt%)	Bismuth:	2.15e+04 kg (2.02 wt%)
Nitrate:	8.74e+04 kg (8.20 wt%)	Iron:	1.89e+04 kg (1.77 wt%)
Phosphate:	5.18e+04 kg (4.87 wt%)	Sulfate:	1.24e+04 kg (1.16 wt%)
Nitrite:	4.79e+04 kg (4.50 wt%)	Silicon:	1.11e+04 kg (1.04 wt%)
Radionuclides in the Waste			
Total Alpha Pu*:	1.07e+02 Ci	Strontium-90:	2.64e+05 Ci
Cesium-137:	1.68e+05 Ci	Total Uranium:	2.10e+02 kg (0.02 wt%)

*Total alpha emitted from ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu.

Since tanks B-110 and B-111 have similar process histories, their sampling results were compared. At the 95% confidence level, there is relatively good agreement between these tanks for six of the major constituents noted in the previous paragraph. The results of the chemical analyses have been compared to the dangerous waste codes in the *Washington Administrative Code*, "Dangerous Waste Regulations" (WAC 173-303). This assessment was conducted by comparing tank analyses against dangerous waste characteristics ("D" waste codes) and against state waste codes. The comparison did not include checking tank analyses against "U", "P", "F", or "K" waste codes since application of these codes is dependent on the source of the waste and not on particular constituent concentrations. The results indicate that the waste in this tank is adequately described in the *Dangerous Waste Permit Application for the Single-Shell Tank System*; this permit is discussed in De Lorenzo et al. (1994).

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LIST OF TERMS

2C	second-decontamination-cycle
A	acid digestion
AA	atomic absorption
ANOVA	analysis of variance
CVAA	cold vapor atomic absorption
D	direct analysis
DL	detection limit
DSC	differential scanning calorimetry
F	KOH/NI fusion
FP	fission product
GC	gas chromatography
GEA	gamma energy analysis
HAS	Hanford Analytical Services
IC	ion chromatography
ICP	inductively coupled plasma
ISE	ion specific electrode
KOH	potassium hydroxide
LANL	Los Alamos National Laboratory
NA	not applicable or not available
NO	not observed
NPH	normal paraffin hydrocarbon
PNNL	Pacific Northwest National Laboratory
PUREX	plutonium-uranium extraction
QA	quality assurance
RCRA	Resource Conservation and Recovery Act
RPD	relative percent difference
RSD	relative standard deviation
SST	single-shell tank
SVOA	semivolatile organic analysis
TC	total carbon
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
TRAC	Track Radioactive Components
VOA	volatile organic analysis
W	water digestion

ACKNOWLEDGEMENTS

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TANK CHARACTERIZATION REPORT FOR
SINGLE-SHELL TANK 241-B-111

1.0 INTRODUCTION

Analysis was conducted on materials obtained from single-shell tank (SST) 241-B-111 (hereafter referred to as B-111) to complete Milestone M-44-05 of the *Hanford Federal Facility Agreement and Consent Order* (Tri-Party Agreement) (Ecology et al. 1994), to sample and analyze two cores from twenty tanks. Measurements taken on the two core samples were used to prepare inventory estimates and to support the following objectives:

- Estimate both the concentration and total quantity of key analytes relating to safety issues, such as organics and radionuclides.

- Provide input to risk-assessment-based decisions regarding disposal of the waste.

- Measure physical properties, such as rheology, bulk density, and particle size.

These measurements and estimates are necessary for the design and fabrication of retrieval, pretreatment, and final waste disposal systems.

1.1 PURPOSE

The purpose of this report is to characterize the waste in SST B-111. "Characterization" includes the determination of the physical, chemical (e.g., concentrations of elements and organic species) and radiological properties of the waste. These determinations are made using analytical results from B-111 core samples together with surveillance and historical information about the tank. The main objective is to determine average waste properties.

This report also consolidates the available historical information regarding tank B-111, arranges the analytical information from the recent core sampling in a useful format, and provides an interpretation of the data within the context of what is known about the tank.

1.2 SCOPE

The waste properties are determined from core samples which were chemically analyzed at the Pacific Northwest National Laboratory (PNNL) Analytical Laboratory

(325-A Laboratory). Additional relevant information on the waste has been compiled from historical sources. Types of historical information that are routinely checked include:

- Past sampling events
- Routine tank surveillance measurements
- Tank transfer records.

This historical information has been reviewed and compared with the laboratory data to help interpret the laboratory data correctly. However, the characterization estimates presented in this report are derived from the laboratory data unless otherwise indicated. It is assumed that the laboratory data provides the most authoritative description of the tank waste.

Since B-111 was not a Watch List tank, relatively few segment-level measurements were performed. This sampling and analysis effort was intended to determine mean concentrations (through composite analysis) in order to meet process design characterization objectives for waste treatment. Process design generally requires knowledge of bulk inventories.

2.0 HISTORICAL TANK INFORMATION

Since 1944, underground storage tanks in Hanford's 200 Areas have been used to store radioactive waste generated by processing plants and laboratories at the Hanford Site. A study of waste management operations records yields information about the process waste types transferred into a tank and the physical state of the waste. Based on the plant effluent stream compositions, transfer records, and the service life history of a tank, a preliminary assessment can be made of the expected waste inventory and its configuration in the tank.

The B Tank Farm is located in the 200 East Area and was constructed during 1943 and 1944 (see Hanford Site Tank Farms diagram for 200 East Area in Hanlon [1993]). The B Tank Farm is one of the original four tank farms (B, C, T, and U) made up of SSTs. There are 16 waste tanks in B Tank Farm. Four tanks (B-201 to B-204) have a nominal capacity of 208,200 L (208 m³). The remaining twelve tanks (B-101 to B-112) have a capacity of 2,006,300 L (2,020 m³).

2.1 TANK DESCRIPTION

A summary of the basic design for tank B-111 is presented in Appendix A. Tank B-111 is one of the 12 large SSTs with a capacity of 2,006,300 L (530,000 gal). The tanks in the tank farms were connected in groups of three or four and overflowed from one to another (known as a cascade). Tank B-111 is the middle tank in a cascade that includes B-110 and B-112. Cascades served several functions in Hanford Site waste management operations. Cascaded tanks require fewer connections to be made during waste disposal; consequently, all three tanks were usable without having to connect the active waste transfer line directly to each individual tank. This handling method reduces the likelihood of personnel being exposed to the waste, and diminishes the chance of a loss of tank integrity due to overfilling. Another benefit of cascading is clarification of the wastes. In a cascade arrangement, most of the solids in the waste slurries routed to the tanks settle in the first tank (B-110), and the clarified liquids cascade on to the other tanks in the series (B-111 and B-112). Supernate from the final tank in the cascade series was sometimes routed to a disposal trench. Since most radionuclides are insoluble in alkaline media, this clarification process reduces the potential radiological contamination of the environment. Tank B-111 is approximately half full, with 897,100 L (237,000 gal) of a sludge type waste.

2.2 PROCESS KNOWLEDGE

The process history for tank B-111 is very similar to that of tank B-110, since much of the waste in tank B-111 came from the cascaded overflow from tank B-110. Tank B-111 received waste from B-110 from 1945 until 1954, when the cascade system was discontinued. Because of their similar process histories, analytical results from core sampling of B-111 should be compared with the core sample results from B-110 and B-112.

Most of the waste in tank B-111 can be characterized as one of two primary waste types: second-decontamination-cycle (2C) waste or fission product (FP) waste. However, other wastes entering tank B-111 are mentioned in Anderson (1990). These other wastes include B Plant cell flush waste, ion exchange waste, and evaporator bottoms waste.

Second-decontamination-cycle waste from the bismuth phosphate process was transferred into tank B-111 from 1945 to 1952 (Anderson 1990). This waste type is expected to contain less than 0.1% of the original fission activity and about 1% of the original plutonium.

Based on historical estimates developed by Los Alamos National Laboratory (LANL) (Brevick 1994), the major constituents in 2C waste are sodium, phosphate, and hydroxide.

Fission product waste generated in the plutonium-uranium extraction (PUREX) process was transferred to B-111 between 1963 and 1967. The PUREX process was used to extract uranium, plutonium, and neptunium from irradiated uranium slugs. In the PUREX process used at Hanford, waste streams (both aqueous and organic) were extensively recycled to the partition cycle; therefore, the primary waste stream from the PUREX process originated from the multistage pulse-column in the partition cycle. This waste stream was concentrated by evaporation and denitrated by sugar addition before the waste was transferred to the underground storage tanks. After concentration and denitration, this PUREX waste stream contained most of the fission products, and is called FP waste. In later years, cesium and strontium were removed from this waste stream prior to its disposal in the underground storage tanks. The major chemical constituents expected in this FP waste type are sodium, iron, hydroxide, and silicate. The most prevalent radionuclide expected is strontium-90.

Based on the history of waste transfers into and out of tank B-111 and the layers observed in the core samples from tank B-110, two distinct waste layers are expected in tank B-111. The bottom layer should be composed of solids which settled from the 2C waste, and the top layer should be composed of the solids which settled from the FP waste.

The estimated composition of the waste in tank B-111 is reported in Table 2-1. Composition estimates from two sources are reported in Table 2-1. The estimates in the second column are derived from the Track Radioactive Components (TRAC) Model (Jungfleisch and Simpson 1993), which is based on tank transfer records and process history. The algorithm employed in TRAC tends to bias the sodium and nitrate contents high. The estimates in the third column of Table 2-1 are derived from a model developed at LANL (Brevick 1994). This model is also based on process history and tank transfer records, but incorporates a larger database of historical records and evaluates the history and transaction records differently than TRAC. No other historical characterization data was found for comparison.

Table 2-1. Estimated Composition of Tank 241-B-111 Contents.

Constituent	TRAC (M)	LANL (M)	LANL (μg/g)
Aluminum	0.223	0.005	105
Bismuth	4.459	0.138	21,900
Carbonate	2.229	0.005	211
Chromium	0.009	0.014	544
Fluoride	0	0.144	2,060
Hydroxide	0.892	1.997	25,700
Iron	0.111	0.524	22,100
Nitrate	33.44	0.776	36,400
Nitrite	0.892	0	0
Phosphate	4.459	1.364	97,900
Potassium	0.033	0	0
Silicate	0.011	0.642	13,600
Sodium	33.44	5.989	104,000
Sulfate	0.892	0.051	3,730
Total organic carbon	NA	NA	152
Uranium	NA	0.062	11,100
	(μCi/g)		(μCi/g)
Cesium-137	657.057	NA	30.6
Plutonium	NA	NA	0.39
Strontium-90	0	NA	1,040
	(g/mL)		(g/mL)
Density	1.8	NA	1.33
	(%)		(%)
Weight percent solids	NA	NA	34.5

2.3 SURVEILLANCE DATA

Each of the 177 underground tanks at the Hanford Site is routinely monitored for supernate levels, solid waste levels, dry well status, and temperature readings. A monthly surveillance report lists the results of this monitoring and the status of each tank (e.g., watch lists, leak status, unusual events).

Figure 2-1 shows the supernate and solids waste levels within tank B-111 from 1945 to the present.[1] Supernate and sludge levels were taken on a quarterly basis as part of the overall surveillance effort in the tank farms. Zero on the vertical scale is at the knuckle bottom of the tank and the dish bottom is below that at -30.48 cm (-12 in.). The sludge level in the tank is indicated by the solid line and the supernate level is indicated by the dashed line. The sludge levels from second quarter 1950 to third quarter 1953 are estimates based on the best engineering interpretation of the historical data. For tank B-111, the early waste level records were not always available on a quarterly basis (Anderson 1990). During these times, it was necessary to estimate the changing surface levels based on best engineering judgement. All of the liquid 2C waste was pumped to a crib in the second quarter of 1950 and again in the second quarter of 1954. The drops in supernate levels shown in the illustration are various transfers out of B-111 to tanks B-108, B-112, and B-103. At present, B-111 contains approximately 893,400 L (236,000 gal) of sludge and approximately 3,800 L (1,000 gal) of supernate. This level is approximately 207.36 cm (81.64 in.) of waste measured at the edge of the tank, and 237.84 cm (93.64 in.) of waste measured at the centerline.

Figure 2-1. Tank Waste-Level Summary for Tank 241-B-111.
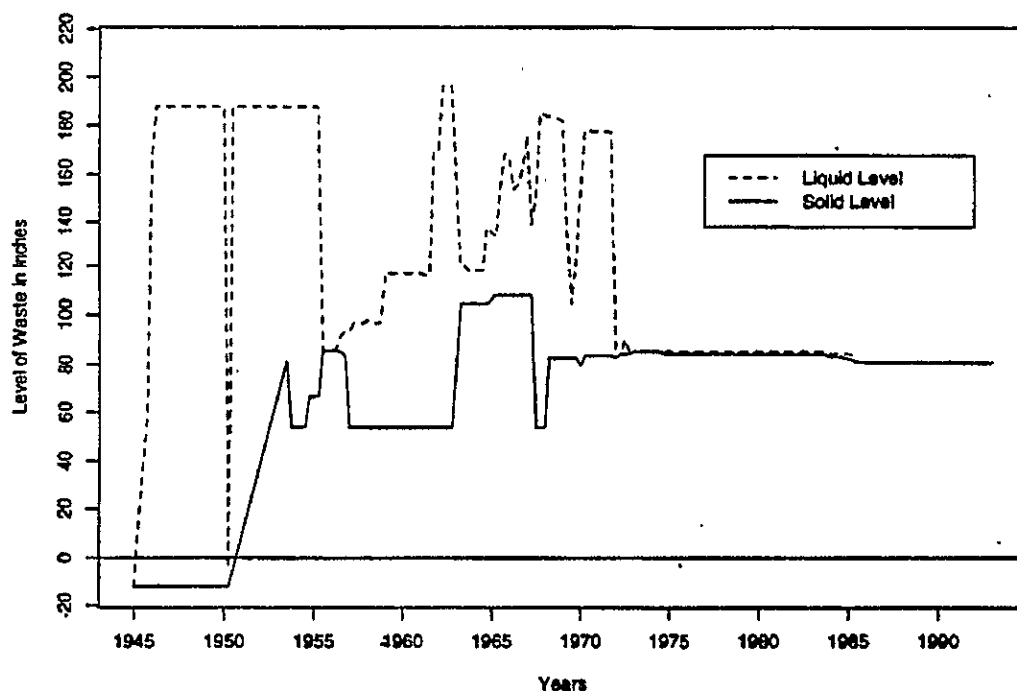
[1]Tank level data were obtained from ICF Kaiser Hanford Company.

2-4

Since 2C waste was the only waste received by B-111 from 1945 to 1952, it is expected that the bottom 121.92 cm (48 in.) of sludge is primarily 2C waste solids. The remaining sludge, above the 121.92 cm (48 in.) mark, is expected to be primarily FP solid waste.

Tank B-111 dome space temperature readings were taken from 1975 to 1990. These readings were taken from a thermocouple tree located at riser 8 (see Appendix A, Figure A-1), containing eleven thermocouples. The mean temperature over this time period was 26.7 °C (80.2 °F), with a standard deviation of 5.5 degrees. The temperature data ranges from 12.5 to 36.6 °C (54.5 to 98 °F). The temperature readings appeared to vary somewhat randomly about the mean over this time period and therefore, conclusions drawn about the temperature readings are limited. This lack of discernible trends can be attributed to the sparse amount of reliable data available for the temperature readings (Hanlon 1993). The 1990 readings are lower and more tightly grouped, with a mean of 12.94 °C (55.3 °F) and a standard deviation of 0.33 degrees. All of the 1990 temperature readings were taken in January. From this examination of temperature history, it is concluded that the observed dome temperatures in tank B-111 are not high enough to warrant concern about high heat evolution.

2.4 TANK STATUS

B-111 is not presently on any watch list and has no unreviewed safety issues associated with it that can be determined from present historical data. B-111 is an interim isolated tank, meaning that all access to the tank not required for long-term surveillance has been sealed in a way that provides at least one barrier to the inadvertent addition of liquid. This tank is also interim stabilized, meaning that as much of the free liquid as possible has been removed with a salt well pump. B-111 was put on the assumed leaker list after an observed drop in the waste surface level (equivalent to approximately 30,300 L or 8,000 gal) in 1978 (Hanlon 1993).

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3.0 TANK SAMPLING OVERVIEW

This section briefly describes the retrieval of tank waste samples from SST B-111. The objective of these procedures is to recover sufficient sample for analytical tests, while maintaining the integrity of any stratification which may exist in the tank. The waste material in SST B-111 is comprised of sludge and liquid. Samples of the waste were obtained by push mode core sampling (see below). Two cores were taken from opposite sides of the tank. The samples were submitted to the analytical laboratory on October 8, 1991, but the laboratory analyses and characterization activities were delayed until February 1993.

3.1 CORE SAMPLING EVENT

The high-level waste tanks in the 200 East and West Area Tank Farms on the Hanford Site are underground storage tanks with a minimum of 1.76 m (6 ft) of soil cover. Because these tanks are underground, access to the waste is limited to existing risers as illustrated in Appendix A, Figure A-1. The underground storage tanks are sampled with specialized core sampling equipment to protect operators and the environment from radiation exposure and contamination. The core sampling equipment is mounted on a truck. The truck is positioned over the desired riser, and a drill string containing the sampler is lowered through the riser into the tank. The truck is equipped with a rotating platform so that the sample can be taken from the tank and the sampler can be remotely placed in a liner and shipping cask. These remote operations reduce the amount of manual handling of the full sampler, thus reducing the radiation dose to which personnel are exposed.

Two types of core samplers (push mode and rotary mode) are currently used in conjunction with the core sampling truck. The push mode sampler is limited to soft materials, while the rotary mode sampler can be used to obtain core samples from harder waste types. Rotary mode sampling requires more time to assemble at the sampling site and safety concerns have been raised about the operation of this sampler (e.g., generation of heat at the drill bit and potential ignition of the waste). These safety concerns have been addressed (Keller 1993), but push mode sampling is generally used whenever possible in order to maintain a conservative safety envelope. Further information about sampling equipment and procedures can be found in De Lorenzo et al. (1994).

Both the push and the rotary mode samplers are constructed of stainless steel. The push mode samplers used to sample tank B-111 were 102 cm (40 in.) long and 3.2 cm (1.25 in.) in diameter, and capture a cylindrical sample 48 cm (19 in.) long and 2.2 cm (7/8 in.) in diameter. The volume of this sample is 187 mL. Once the sampler is lowered through the drill string to the appropriate depth for sampling, a piston inside the cylindrical sample reservoir is held stationary as the sampler is pushed through the waste. The 5.08 cm (2 in.) diameter drill string is fitted with a blunt drill bit which cuts the waste and directs it into the sampler. Tank stratification is maintained in the sample, since the sample is not

pulled or poured into the sampler. The sample is captured in the sampler by a rotary valve which is closed when the sampler has been pushed 48 cm (19 in.). The closed sampler is extracted from the drill string and another sampler is inserted. The drill string is then lowered another 48 cm (19 in.) to capture the next segment of waste. A complete core sample consists of as many 48 cm (19 in.) segments as are needed to sample the depth of the waste in the tank (Giamberardini 1993).

After a segment is captured by the sampler, it is sealed within a stainless steel liner and placed in a shipping cask. The casks are transported to the analytical laboratory for sample identification, storage, and analysis. The five segments of material recovered from riser 3 constitute core 29. Five segments of material were also recovered and extruded from riser 5 on the opposite side of the tank, and these five segments constitute core 30.

As shown in Table 3-1, segment 1 was not recovered for either core. For core 29, segments 2 through 5 were completely recovered. For core 30, segments 3 and 4 were completely recovered, and segments 2 and 5 were only partially recovered.

Table 3-1. Actual Percent Recovery in Tank 241-B-111.

	Core 29 (Riser 3)	Core 30 (Riser 5)
1	0%	0%
2	100%	16%
3	100%	100%
4	100%	100%
5	100%	35%

After extrusion from the sampler, the core material was placed in glass bottles, sealed and stored in the High-Level Radioactive Facility. Laboratory analysis and characterization activities were delayed until February 1993 because analytical work on tank SY-101 and the Ferrocyanide Safety Program took precedence.

3.2 ADDITIONAL TANK SAMPLING

No other sampling information is available on tank B-111.

4.0 SAMPLE HANDLING AND ANALYTICAL SCHEME

The sample handling, sample preparation, and types of analysis performed on the samples are described in this section.

4.1 WASTE DESCRIPTION

The two cores recovered from tank B-111, core 29 and core 30, were very similar except that drainable liquid was contained only in core 30. Both cores were sludges that held their shape upon extrusion. The flow behavior and lower density of the solids in core 30 indicated that there was some mixing of the solid material and drainable liquid. The sample color in both cores varied from dark brown to tan.

The drainable liquid contained in segments 2 and 5 from core 30 was normal paraffin hydrocarbon (NPH). This drainable liquid had a density of 0.80 g/mL and appeared to be organic. The density and appearance of the liquid is consistent with the properties of NPH, but it was not analyzed. NPH is the hydrostatic drilling fluid used for this sampling event.

As shown in Table 3-1, four segments of core 29 were fully recovered. Each of these segments weighed about 230 g. Two segments of core 30 were fully recovered, and two were partially recovered. Segment 2 from core 30 contained 140 mL of drainable liquid and only 30 mL or 38 g of solids, which represents 16% of the expected volume of solids. Segment 5 from core 30 contained 65 mL of drainable liquid and only 70 mL or 87 g of solids, which represents 37% of the expected volume of solids. There is no notation of mechanical failure to account for the partial recoveries of these samples. However, judging from the amount of liquid captured in the sampler, there appears to have been an incomplete seal around the sampler opening during the sampling which allowed liquid (either hydrostatic head fluid or drainable liquid) into the sampler, impeding operations.

Each segment from both cores was photographed in the extrusion tray. Figure 4-1 shows the segments for core 29 and Figure 4-2 shows the segments for core 30. For core 29, segments 2 through 5 are labeled 91-081 through 91-084, respectively. For core 30, segments 2 through 5 are labeled 90-086 through 90-089, respectively.

4.2 HOLDING TIME CONSIDERATIONS

All analyses have limits imposed between the time a sample is recovered and the time of analysis (hold time limitations). No attempt was made to meet holding time limits for these samples due to waste disposal issues and program priorities. The samples were received on October 8, 1991, and analysis commenced in February 1993.

Figure 4-1. Segment Photographs for Core 29.

Figure 4-2. Segment Photographs for Core 30.
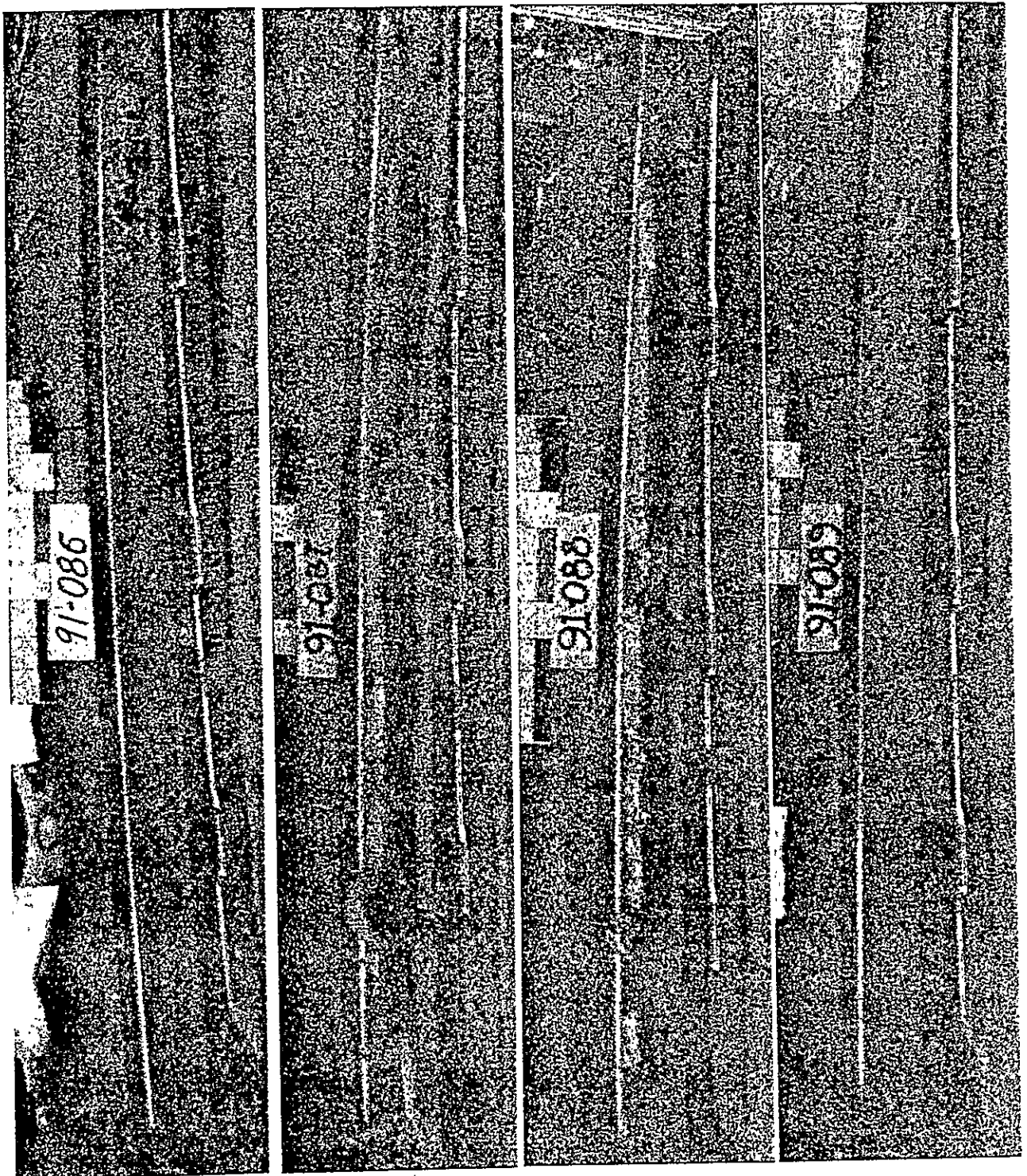
4.3 SAMPLE PREPARATION AND ANALYTICAL METHODS

Figure 4-3 is a flowchart of the steps taken by the 325-A Laboratory to analyze tank core samples. The B-111 core samples were received from Westinghouse Hanford Company tank farms personnel and were extruded at PNNL's Hot Cell Facility, the 325-A Laboratory. Segment photographs were taken, aliquots were extracted from each segment for volatile organics analysis (VOA), and physical property assays (e.g., particle size) were performed. The segments were homogenized, and a limited number of homogenization test samples were taken (homogenization test results are detailed in Sections 4.4 and 7.0). Composite samples were created from the homogenized aliquots, and the procedure was repeated to develop independent duplicate composites for each core. Generally, additional homogenization test samples are taken from the composite samples. But this was not done for cores 29 and 30 composite samples from tank B-111. After some investigation, no reason has been found as to why homogenization tests were not performed on the composites.

Figure 4-3. Data Collection and Preparation.
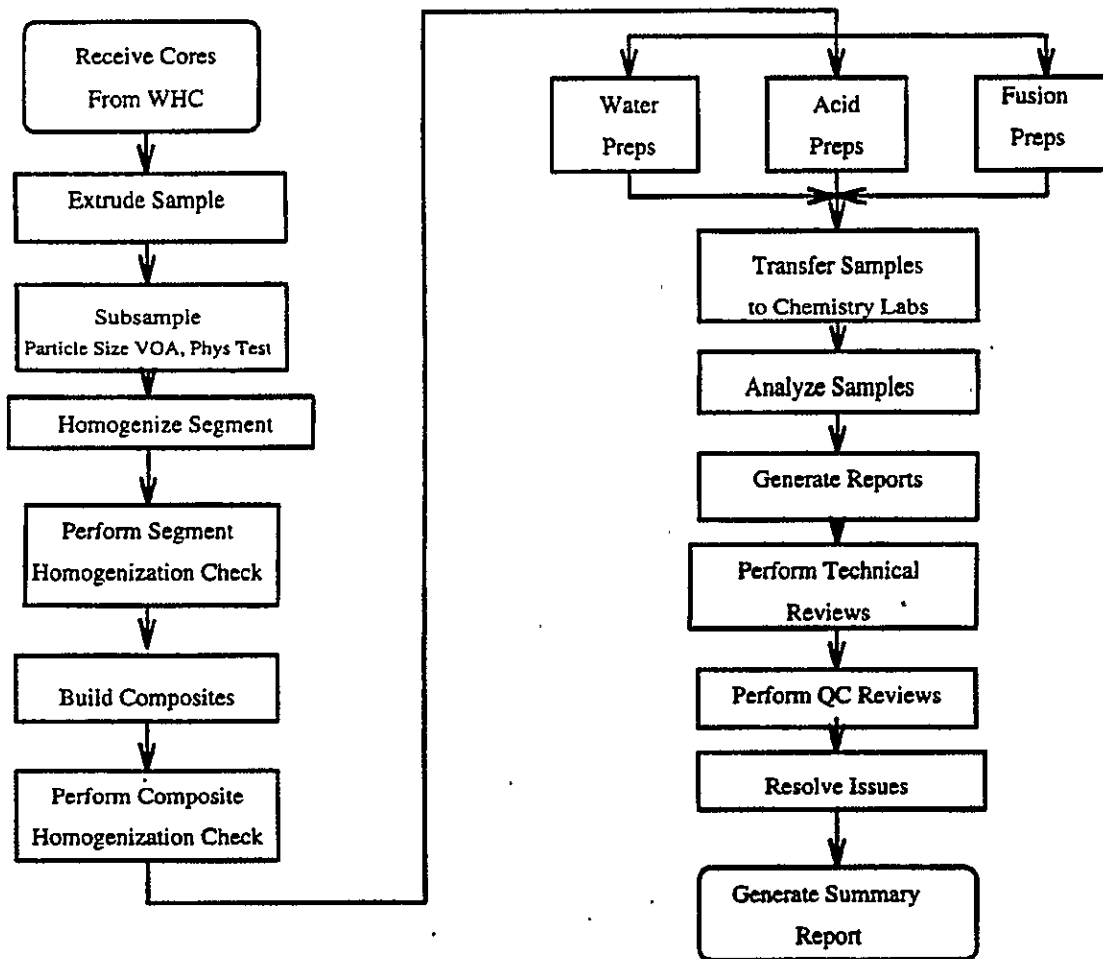
Caustic fusion, acid digestion, and water leach preparations of all core composites were completed in the Shielded Analytical Laboratory. Tests requiring little or no sample preparation, such as weight percent solids, direct total carbon, direct total inorganic carbon, direct total organic carbon, carbon-14, and pH, were conducted in-cell. Because of the low level of radioactivity of the sample material, aliquots were provided directly to the 325-A Laboratory for mercury, toxicity characterization leach procedure, semivolatile organic analysis (SVOA), and extraction organic halides analysis.

The Shielded Analytical Laboratory made deliberate minor deviations to sample preparation procedures for one or more of the following reasons:

- Insufficient sample was available to conduct the analysis according to the specified procedure, and still maintain the level of quality control requested.

- Sample weights and/or final volumes were reduced to comply with waste minimization requirements.

- Sample weights and/or final volumes were altered to increase the concentration of certain analytes of interest. This was done to meet the concentration ranges needed to perform the analysis, as specified in the procedures.

These deviations are not expected to have a substantive impact on the analytical results or on any conclusions derived from those results. Table 4-1 lists the sample preparation and analytical methods used to obtain analyte concentration estimates for B-111 samples. The preferred methods, those methods expected to yield the most valid analytical results for waste inventory calculation, are given in Table 4-1. After the samples were chemically analyzed, laboratory core reports were generated and reviewed. After the review process was finished and various issues were resolved, a final summary report was issued (Giamberardini 1993).

Table 4-1. Sample Preparation and Analytical Methods
Used on Tank 241-B-111 Samples. (2 sheets)

Analyte	Sample Prep.	Preferred Method	Analyte	Sample Prep.	Preferred Method
Aluminum	A,F,W	ICP:A	Antimony	A,F,W	ICP:A
Arsenic	A,F,W	ICP:A	Barium	A,F,W	ICP:A
Bismuth	A,F,W	ICP:F	Beryllium	A,F,W	ICP:A
Boron	A,F,W	ICP:A	Cadmium	A,F,W	ICP:A
Calcium	A,F,W	ICP:A	Cerium	A,F,W	ICP:A
Chromium	A,F,W	ICP:A	Cobalt	A,F,W	ICP:A
Copper	A,F,W	ICP:A	Dysprosium	A,F,W	ICP:A
Europium	A,F,W	ICP:A	Gadolinium	A,F,W	ICP:A
Iron	A,F,W	ICP:F	Lanthanum	A,F,W	ICP:A
Lead	A,F,W	ICP:A	Lithium	A,F,W	ICP:A
Magnesium	A,F,W	ICP:A	Manganese	A,F,W	ICP:A
Molybdenum	A,F,W	ICP:A	Neodymium	A,F,W	ICP:A
Nickel	A,F,W	ICP:A	Palladium	A,F,W	ICP:A
Phosphorus	A,F,W	ICP:F	Potassium	A,F,W	ICP:A
Rhodium	A,F,W	ICP:A	Ruthenium	A,F,W	ICP:A
Selenium	A,F,W	ICP:A	Silicon	A,F,W	ICP:F
Silver	A,F,W	ICP:A	Sodium	A,F,W	ICP:F
Strontium	A,F,W	ICP:A	Tellurium	A,F,W	ICP:A
Thallium	A,F,W	ICP:A	Thorium	A,F,W	ICP:A
Tin	A,F,W	ICP:A	Titanium	A,F,W	ICP:A
Tungsten	A,F,W	ICP:A	Vanadium	A,F,W	ICP:A
Yttrium	A,F,W	ICP:A	Zinc	A,F,W	ICP:A
Zirconium	A,F,W	ICP:A	Chloride	W	IC:W
Cyanide	W	IC:W	Fluoride	W	IC:W
Nitrate	W	IC:W	Nitrite	W	IC:W
Phosphate	W	IC:W	Sulfate	W	IC:W
Ammonia	W	ISE:W	Mercury	A	CVAA:A
Curium-243/244	F	Alpha Radchem:F	Gross alpha	F	Alpha Radchem:F
Neptunium-237	F	Alpha Radchem:F	Plutonium-238	F	Alpha Radchem:F
Plutonium-239/240	F	Alpha Radchem:F	Total alpha	F,W	Alpha Radchem:F
Gross beta	F,W	Beta Radchem:F	Strontium-90	F	Beta Radchem:F
Technetium-99	F	Beta Radchem:F	Americium-241	A,F,W	GEA:F
Cerium-144	A,F,W	GEA:F	Cesium-134	A,F,W	GEA:F
Cesium-137	A,F,W	GEA:F	Cobalt-60	A,F,W	GEA:F
Europium-154	A,F,W	GEA:F	Europium-155	A,F,W	GEA:F

Table 4-1. Sample Preparation and Analytical Methods
Used on Tank 241-B-111 Samples. (2 sheets)

Analyte	Sample Prep.	Preferred Method	Analyte	Sample Prep.	Preferred Method
Potassium-40	A,F,W	GEA:F	Uranium	F	Laser Fluorimetry:F
Plutonium-239	F	Mass Spectrometry:F	Plutonium-240	F	Mass Spectrometry:F
Plutonium-241	F	Mass Spectrometry:F	Plutonium-242	F	Mass Spectrometry:F
Uranium-234	F	Mass Spectrometry:F	Uranium-235	F	Mass Spectrometry:F
Uranium-236	F	Mass Spectrometry:F	Uranium-238	F	Mass Spectrometry:F
Tritium	W	Liq. Scintillation:W	Carbon-14	W	Liq. Scintillation:W
Nickel-59	A	Liq. Scintillation:A	Nickel-63	A	Beta Radchem:A
TOC	D,W	Persulfate Oxidation:W	Hex. Chromium	W	Calorimetric:W
Total carbon	D,W	Persulfate Oxidation:W	TIC	D,W	Persulfate Oxidation:W
SVOA		GC/Mass Spectrometry	VOA		GC/Mass Spectrometry

4.4 SAMPLE HOMOGENEITY

The eight segments from cores 29 and 30 were individually homogenized, as mentioned in the previous section. Segment 4 from core 29 and segments 3 and 5 from core 30 were subsampled for the homogenization tests. These subsamples were prepared for analysis by caustic fusion and submitted to the laboratory for gamma energy analysis (GEA), inductively coupled plasma (ICP) analysis, and total alpha analysis. The results of this homogenization test are discussed in Section 7.2.

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5.0 ANALYTICAL RESULTS AND WASTE INVENTORY

A total of 4,625 analytical measurements were made on tank B-111; Table 5-1 contains a summary of the analytical result counts. As shown, the most complete segment-level analyses were performed on physical properties. All of the segment-level chemical analyses were homogenization tests. Nearly one-third of all analytical results in the B-111 dataset are quality assurance (QA) data (i.e., matrix spikes, method blanks, etc.). If the homogenization test data are included as QA data, this percentage increases to 45% (i.e., almost one-half of the analytical results in the B-111 dataset are QA data).

Table 5-1. Summary of Tank 241-B-111 Analytical Result Counts.

		Segment					Composite	Totals
		1	2	3	4	5		
Physical properties	Core 29	0	42	47	50	55	6	200
	Core 30	0	48	50	46	49	6	199
Chemical analyses	Core 29	0	0	0	196	0	1,096	1,292
	Core 30	0	0	196	0	196	1,063	1,455
Quality assurance data		0	0	0	49	49	1,381	1,479
Totals		0	90	293	341	349	3,552	4,625

The core composite data was used to determine mean concentrations and their associated uncertainties. These values were then used to estimate the waste inventory of tank B-111. The available segment-level data was used to conduct the sample homogenization tests and to determine the physical properties of tank B-111 waste. A summary of the results from the statistical analysis is given in this section. The complete results are contained in Appendix B.

5.1 CHEMICAL ANALYSES AND RADIOLOGICAL DETERMINATIONS

As a result of the sampling structure in the B-111 composite data, the following random effects model was fit to describe the mean concentration and variability of each constituent:

$$Y_{ijk} = \mu + C_i + S_{ij} + E_{ijk} \tag{1}$$

where:

Y_{ijk} = The measured value of concentration of a constituent in replicate j of core i

μ = The mean concentration of the constituent

C_i = The deviation of concentration in core i from the mean value

S_{ij} = The deviation of concentration in core replicates from the mean value (two replicates were processed on each composite)

E_{ijk} = The analytical (lab) error in the measurement.

As one can see, each term in the model describes the contribution to the variability of a step in the sampling and measurement process. For each constituent, this model can be used to obtain a mean concentration estimate along with its associated uncertainty. This model can also be used to obtain estimates of horizontal variability (σ_C^2), sampling variability (σ_S^2), and analytical variability (σ_E^2) for each constituent.

Table 5-2 shows the results of fitting the random effects model of Equation 1 for each constituent. The estimated mean concentration, its associated relative standard deviation (RSD),[2] and total inventory are given for each constituent. If more than 75% of the sample results for a given constituent were below the detection limit, the random effects model was not fit. In that case, a mean of the detection limits was reported and RSDs were not calculated. Some of the constituents shown in this table were analyzed by more than one method, but only the results from the preferred analytical method are presented. The complete set of constituent results (for all constituents and analytical methods), including the individual variance component estimates, is contained in Appendix B.

The boxplots in Figure 5-1 illustrate the magnitude of horizontal, sampling, and analytical variance components relative to each other. The "box" for a given boxplot represents the range of the middle 50% of the RSDs. The vertical line in each box is the median RSD value and the lines (whiskers) emanating from the ends of the boxes represent the entire range of the RSDs. For all subgroupings of constituents (anions, metals, organics, radionuclides), the horizontal spatial variability is generally the largest source of variability. For the cations, the longest whisker on the horizontal variability boxplot is due to the copper acid digestion ICP analysis (see Appendix B).

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[2]The RSD is the square root of the variance estimate divided by the estimated mean of the constituent, which indicates how large the variance estimate is relative to the mean.

Table 5-2. Summary of the Composite Level Results for Anions, Metals, Organics and Radionuclides. (5 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration			Total Inventory
		Composite	RSD	Hist.	
Anions		(μg/g)			(kg)
Chloride	IC:W	1.02e+03	2	NA	1.09e+03
Cyanide	CN:W	1.88e+00	19	NA	2.00e+00
Fluoride	IC:W	1.56e+03	2	2.06e+03	1.66e+03
Nitrate	IC:W	8.20e+04	8	3.64e+04	8.74e+04
Nitrite	IC:W	4.50e+04	9	0.00e+00	4.79e+04
Phosphate	IC:W	2.39e+04	3	9.79e+04	2.55e+04
Phosphate	ICP:F	4.87e+04	8	9.79e+04	5.18e+04
Sulfate	IC:W	1.16e+04	1	3.73e+03	1.24e+04
Cations		(μg/g)			(kg)
Aluminum	ICP:A	8.99e+02	7	1.05e+00	9.58e+02
Ammonia	ISE:W	4.58e+01	38	NA	4.88e+01
Antimony	ICP:A	1.83e+01	28	NA	1.95e+01
Arsenic	ICP:A	2.79e+01	NA	NA	2.97e+01
Barium	ICP:A	2.82e+01	11	NA	3.00e+01
Beryllium	ICP:A	<1.74e+00	NA	NA	<1.85e+00
Bismuth	ICP:F	2.02e+04	1	2.19e+04	2.15e+04
Boron	ICP:A	5.14e+01	7	NA	5.48e+01
Cadmium	ICP:A	2.77e+00	15	NA	2.95e+00
Calcium	ICP:A	6.89e+02	23	NA	7.34e+02
Cerium	ICP:A	3.21e+01	24	NA	3.42e+01
Chromium	ICP:A	1.11e+03	5	5.44e+00	1.18e+03
Cobalt	ICP:A	4.43e+00	21	NA	4.72e+00
Copper	ICP:A	2.01e+02	94	NA	2.14e+02
Dysprosium	ICP:A	<6.97e+00	NA	NA	<7.43e+00
Europium	ICP:A	<3.49e+00	NA	NA	<3.72e+00
Gadolinium	ICP:A	<6.97e+01	NA	NA	<7.43e+01
Hexavalent Chromium	Calorimetric:W	1.61e+02	6	NA	1.72e+02
Iron	ICP:F	1.77e+04	5	2.21e+04	1.89e+04
Lanthanum	ICP:A	1.13e+01	27	NA	1.20e+01
Lead	ICP:A	1.57e+03	7	1.33e-01	1.67e+03
Lithium	ICP:A	<6.97e+00	NA	NA	<7.43e+00
Magnesium	ICP:A	1.95e+02	2	NA	2.08e+02
Manganese	ICP:A	7.89e+01	6	0.00e+00	8.41e+01
Mercury	CVAA(Hg):A	9.32e+00	50	NA	9.93e+00

Table 5-2. Summary of the Composite Level Results for Anions, Metals, Organics and Radionuclides. (5 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration			Total Inventory
		Composite	RSD	Hist.	
Molybdenum	ICP:A	4.17e+01	9	NA	4.44e+01
Neodymium	ICP:A	2.21e+01	23	NA	2.35e+01
Nickel	ICP:A	2.07e+01	7	NA	2.21e+01
Palladium	ICP:A	5.25e+01	NA	NA	5.59e+01
Phosphorus	ICP:F	1.59e+04	8	NA	1.69e+04
Potassium	ICP:A	6.74e+02	18	0.00e+00	7.18e+02
Rhodium	ICP:A	<3.49e+01	NA	NA	<3.72e+01
Ruthenium	ICP:A	<1.74e+01	NA	NA	<1.85e+01
Selenium	ICP:A	3.23e+01	22	NA	3.44e+01
Silicon	ICP:F	1.04e+04	8	NA	1.11e+04
Silver	ICP:A	5.95e+00	26	NA	6.34e+00
Sodium	ICP:F	9.57e+04	2	1.04e+05	1.02e+05
Strontium	ICP:A	2.18e+02	2	NA	2.32e+02
Tellurium	ICP:A	3.60e+01	28	NA	3.84e+01
Thallium	ICP:A	<1.74e+02	NA	NA	<1.85e+02
Tin	ICP:A	<2.79e+02	NA	NA	<2.97e+02
Titanium	ICP:A	7.90e+00	14	NA	8.42e+00
Tungsten	ICP:A	<2.79e+01	NA	NA	<2.97e+01
Uranium	Laser Fluorimetry:F	1.97e+02	4	NA	2.10e+02
Vanadium	ICP:A	3.93e+00	25	NA	4.19e+00
Yttrium	ICP:A	3.93e+00	25	NA	4.19e+00
Zinc	ICP:A	1.11e+02	50	NA	1.18e+02
Zirconium	ICP:A	1.44e+01	29	NA	1.53e+01
Organics		(µg/g)			(kg)
1,2,4-Trichlorobenzene	SVOA	<9.61e+00	NA	NA	<1.02e+01
1,2-Dichlorobenzene	SVOA	<9.61e+00	NA	NA	<1.02e+01
1,3-Dichlorobenzene	SVOA	<9.61e+00	NA	NA	<1.02e+01
1,4-Dichlorobenzene	SVOA	<9.61e+00	NA	NA	<1.02e+01
2,4,5-Trichlorophenol	SVOA	<4.81e+01	NA	NA	<5.12e+01
2,4,6-Trichlorophenol	SVOA	<9.61e+00	NA	NA	<1.02e+01
2,4-Dichlorophenol	SVOA	<9.61e+00	NA	NA	<1.02e+01
2,4-Dimethylphenol	SVOA	<9.61e+00	NA	NA	<1.02e+01
2,4-Dinitrophenol	SVOA	<4.81e+01	NA	NA	<5.12e+01
2,4-Dinitrotoluene	SVOA	<9.61e+00	NA	NA	<1.02e+01
2,6-Dinitrotoluene	SVOA	<9.61e+00	NA	NA	<1.02e+01

Table 5-2. Summary of the Composite Level Results for Anions, Metals, Organics and Radionuclides. (5 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration			Total Inventory
		Composite	RSD	Hist.	
2-Chloronaphthalene	SVOA	<9.61e+00	NA	NA	<1.02e+01
2-Chlorophenol	SVOA	<9.61e+00	NA	NA	<1.02e+01
2-Methylnaphthalene	SVOA	<9.61e+00	NA	NA	<1.02e+01
2-Methylphenol	SVOA	<9.61e+00	NA	NA	<1.02e+01
2-Nitroaniline	SVOA	<4.81e+01	NA	NA	<5.12e+01
2-Nitrophenol	SVOA	<9.61e+00	NA	NA	<1.02e+01
3,3-Dichlorobenzidine	SVOA	<1.94e+01	NA	NA	<2.07e+01
3-Nitroaniline	SVOA	<4.81e+01	NA	NA	<5.12e+01
4,6-Dinitro-o-cresol	SVOA	<4.81e+01	NA	NA	<5.12e+01
4-Bromophenylphenyl ether	SVOA	<9.61e+00	NA	NA	<1.02e+01
4-Chloro-3-methylphenol	SVOA	<9.61e+00	NA	NA	<1.02e+01
4-Chloroaniline	SVOA	<9.61e+00	NA	NA	<1.02e+01
4-Chlorophenylphenyl ether	SVOA	<9.61e+00	NA	NA	<1.02e+01
4-Methylphenol	SVOA	<9.61e+00	NA	NA	<1.02e+01
4-Nitroaniline	SVOA	<4.81e+01	NA	NA	<5.12e+01
4-Nitrophenol	SVOA	<4.81e+01	NA	NA	<5.12e+01
Acenaphthene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Acenaphthylene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Anthracene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Benzo(a)anthracene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Benzo(a)pyrene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Benzo(b)fluoranthene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Benzo(ghi)perylene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Benzo(k)fluoranthene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Benzoic acid	SVOA	<4.81e+01	NA	NA	<5.12e+01
Benzyl alcohol	SVOA	<9.61e+00	NA	NA	<1.02e+01
Bis(2-Chloroethoxy)methane	SVOA	<9.61e+00	NA	NA	<1.02e+01
Bis(2-chloroethyl) ether	SVOA	<9.61e+00	NA	NA	<1.02e+01
Bis(2-chloroisopropyl)	SVOA	<9.61e+00	NA	NA	<1.02e+01
Bis(2-ethylhexyl) phthalate	SVOA	2.73e+00	8	NA	2.91e+00
Butylbenzylphthalate	SVOA	<9.61e+00	NA	NA	<1.02e+01
Chrysene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Decane	SVOA	1.68e+01	16	NA	1.79e+01
Di-n-butylphthalate	SVOA	8.44e+00	NA	NA	8.99e+00
Di-n-octylphthalate	SVOA	<9.61e+00	NA	NA	<1.02e+01

Table 5-2. Summary of the Composite Level Results for Anions, Metals,
Organics and Radionuclides. (5 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration			Total Inventory
		Composite	RSD	Hist.	
Dioctyl adipate	SVOA	1.20e+01	17	NA	1.28e+01
Dibenz[a,h]anthracene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Dibenzofuran	SVOA	<9.61e+00	NA	NA	<1.02e+01
Diethylphthalate	SVOA	<9.61e+00	NA	NA.	<1.02e+01
Dimethyl phthalate	SVOA	<9.61e+00	NA	NA	<1.02e+01
Dodecane	SVOA	7.96e+02	68	NA	8.48e+02
Extractable total organic halides	Ext Org Halides	<1.00e+01	NA	NA	<1.07e+01
Fluoranthene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Fluorene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Hexachlorobenzene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Hexachlorobutadiene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Hexachlorocyclopentadiene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Hexachloroethane	SVOA	<9.61e+00	NA	NA	<1.02e+01
Indeno(1,2,3-cd)pyrene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Isophorone	SVOA	<9.61e+00	NA	NA	<1.02e+01
N-Nitroso-di-n-dipropylamine	SVOA	<9.61e+00	NA	NA	<1.02e+01
N-Nitrosodiphenylamine	SVOA	<9.61e+00	NA	NA	<1.02e+01
Naphthalene	SVOA	9.61e+00	NA	NA	1.02e+01
Nitrobenzene	SVOA	9.61e+00	NA	NA	1.02e+01
Pentachlorophenol	SVOA	4.81e+01	NA	NA	5.12e+01
Pentadecane	SVOA	5.50e+01	60	NA	5.86e+01
Phenanthrene	SVOA	9.61e+00	NA	NA	1.02e+01
Phenol	SVOA	9.61e+00	NA	NA	1.02e+01
Pyrene	SVOA	<9.61e+00	NA	NA	<1.02e+01
Tetradecane	SVOA	1.14e+03	49	NA	1.21e+03
Total carbon	Persulfate Oxidation:W	5.34e+03	7	NA	5.69e+03
Total inorganic carbon	Persulfate Oxidation:W	4.46e+03	11	NA	4.75e+03
Total organic carbon	Persulfate Oxidation:W	8.75e+02	12	1.52e+02	9.32e+02
Tributyl phosphate	SVOA	2.20e+01	14	NA	2.34e+01
Tridecane	SVOA	1.73e+03	54	NA	1.84e+03
Undecane	SVOA	3.55e+01	15	NA	3.78e+01
Physical Properties					
pH Measurement	pH:W	8.87e+00	1	NA	NA
		(%)			
Weight percent solids	Percent Solid:D	3.69e+01	2	NA	NA

Table 5-2. Summary of the Composite Level Results for Anions, Metals, Organics and Radionuclides. (5 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration			Total Inventory
		Composite	RSD	Hist.	
Radionuclides		(µCi/g)			(Ci)
Americium-241	GEA:F	8.46e-02	25	NA	9.01e+01
Carbon-14	Liquid Scintillation:W	1.60e-03	36	NA	1.70e+00
Cesium-137	GEA:F	1.58e+02	9	3.06e+01	1.68e+05
Cobalt-60	GEA:F	<3.87e-03	NA	NA	<4.12e+00
Curium-242	Alpha Radchem:F	9.16e-05	29	NA	9.76e-02
Curium-243/244	Alpha Radchem:F	4.70e-04	57	NA	5.01e-01
Europium-154	GEA:F	1.70e-01	26	NA	1.81e+02
Europium-155	GEA:F	2.00e-01	30	NA	2.13e+02
Gross alpha	Alpha Radchem:F	1.76e-01	6	NA	1.88e+02
Gross beta	Beta Radchem:F	6.28e+02	15	NA	6.69e+05
Neptunium-237	Alpha Radchem:F	7.14e-05	22	NA	7.61e-02
Plutonium-238	Alpha Radchem:F	.3.05e-03	10	NA	3.25e+00
Plutonium-239/240	Alpha Radchem:F	9.73e-02	5	NA	1.04e+02
Strontium-90	Beta Radchem:F	2.48e+02	22	1.04e+03	2.64e+05
Technetium-99	Beta Radchem:F	1.14e-01	10	NA	1.21e+02
Thorium-232	ICP:A	<2.79e+02	NA	NA	<2.97e+02
Total alpha Pu*	Alpha Radchem:F	1.00e-01	5	NA	1.07e+02
Tritium	Liquid Scintillation:W	2.75e-03	15	NA	2.93e+00
		(%)			
Uranium-234	Mass Spectrometry:F	5.27e-03	7	NA	NA
Uranium-235	Mass Spectrometry:F	6.62e-01	0	NA	NA
Uranium-236	Mass Spectrometry:F	9.35e-03	5	NA	NA
Uranium-238	Mass Spectrometry:F	9.93e+01	0	NA	NA

*Total alpha emitted from Pu-238, Pu-239, Pu-240, Pu-241.

Figure 5-1. Relative Standard Deviation Distributions for Variance Components
Calculated from the Composite-Level Data.
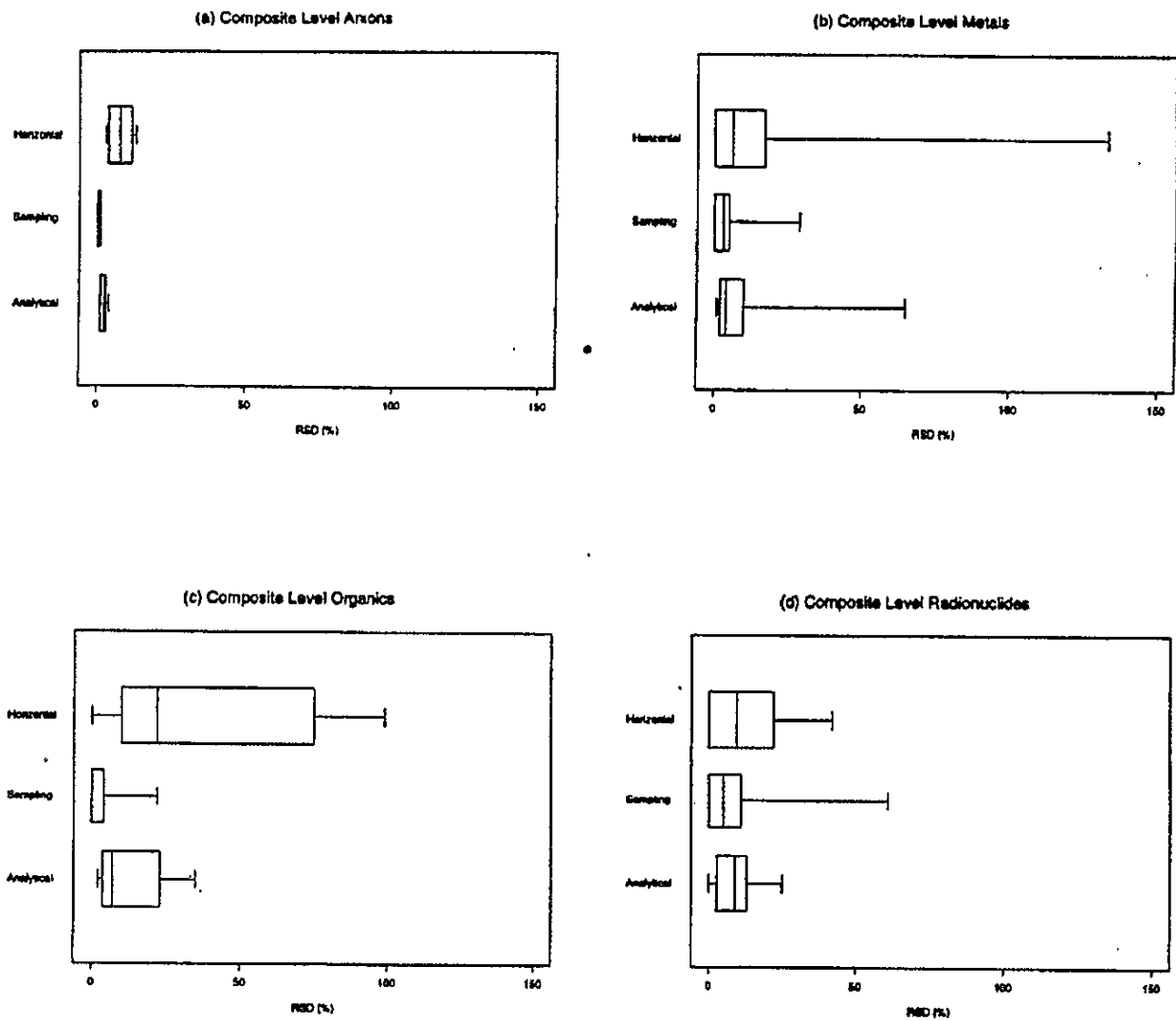
Table 5-3 lists several of the constituents in groups, according to the core in which they were found in highest concentration. Constituents were grouped with one core or the other only if the differences between core results were greater than the uncertainty due to sampling and analytical error. For the nine constituents with the highest concentrations in the B-111 core samples (i.e., those with mean concentrations greater than 10,000 ppm using the preferred analytical method), there was no readily apparent pattern in the results. These nine analytes are denoted by asterisks in Table 5-3. Of these nine analytes, six had mean concentrations which were significantly greater in core 30 than in core 29, while two (sodium and nitrite) had a greater mean concentration in core 29. The remaining major constituent (bismuth) showed no significant statistical difference in the mean values between the two cores. These differences were determined using an analysis of variance (ANOVA). Constituents were excluded from this analysis (i.e., no ANOVA was run) if 75% or more of the sample and duplicate results were below the detection limit.

Table 5-3. Analytes Grouped According to Concentration Differences Between Cores.

Analytes with higher concentrations for Core 29		
Curium-242	Gross alpha	Neptunium-237
Gross beta	Strontium-90	Technetium-99
Americium-241	Cesium-137	Europium-154
Europium-155	Barium	Manganese
Molybdenum	Titanium	Yttrium
Zirconium	Sodium*	Nitrite*
Uranium	Uranium-236	Total carbon
Total inorganic carbon	pH measurement	

Analytes with no statistical differences between cores		
Hexavalent Chromium	Curium-243/244	Plutonium-238
Plutonium-239/240	Total alpha	Antimony
Boron	Cadmium	Cerium
Cobalt	Lanthanum	Magnesium
Neodymium	Nickel	Potassium
Selenium	Silver	Strontium
Tellurium	Vanadium	Bismuth*
Chloride	Carbon-14	Uranium-234
Uranium-235	Uranium-238	Bis(2-ethylhexyl) phthalate
Total organic carbon		

Analytes with higher concentrations for Core 30		
Aluminum	Calcium	Chromium
Copper	Lead	Zinc
Iron*	Phosphorus*	Silicon*
Fluoride	Nitrate*	Phosphate*
Tritium	Weight percent solids	Ammonia
Dioctyl adipate	Dodecane	Pentadecane
Tetradecane	Tridecane	Sulfate*

*Major constituents (>10,000 ppm)

Table 5-4 contains several potentially anomalous results that were noted in the ANOVA residual plots. These results were noted because of their large disagreement with the other results for the particular constituent. The core 29 and core 30 laboratory reports (Giamberardini 1993) were consulted in order to understand why these results were so different from the other results. The information from the core reports is discussed in the paragraphs that follow.

Table 5-4. Composite Values Flagged as Suspect.

	Core	Composite	Aliquot	Value	Below DL	Units
Antimony:ICP:A	29	1	1	49.8060	yes	µg/g
Antimony:ICP:A	30	2	1	47.4700	yes	µg/g
Boron:ICP:F	30	1	2	186.0000	no	µg/g
Cadmium:ICP:F	29	1	1	41.0000	no	µg/g
Calcium:ICP:W	29	1	1	45.8450	yes	µg/g
Carbon-14:Liquid Scintillation:W	29	2	1	0.0181	no	µCi/g
Carbon-14:Liquid Scintillation:W	30	2	1	0.0280	no	µCi/g
Carbon-14:Liquid Scintillation	30	1	1	0.0053	no	µCi/g
Cerium:ICP:A	29	1	1	79.6896	yes	µg/g
Cerium:ICP:A	30	2	1	75.9520	yes	µg/g
Curium-243/244:Alpha Radchem:F	30	1	2	0.0020	no	µCi/g
Lanthanum:ICP:A	29	1	1	29.8836	yes	µg/g
Lanthanum:ICP:A	30	2	1	28.4820	yes	µg/g
Lead:ICP:W	29	1	1	55.0140	yes	µg/g
Mercury:CVAA (Hg):A	30	2	1	19.0000	no	µg/g
Neodymium:ICP:W	29	1	1	27.5070	yes	µg/g
Nickel:ICP:A	29	1	1	29.8836	yes	µg/g
Nickel:ICP:A	30	2	1	28.4820	yes	µg/g
Potassium:ICP:W	29	1	1	916.9000	yes	µg/g
Selenium:ICP:A	29	1	1	74.7090	yes	µg/g
Selenium:ICP:A	30	2	1	71.2050	yes	µg/g
Strontium:ICP:W	29	1	1	4.5845	yes	µg/g
Tellurium:ICP:A	29	1	1	99.6120	yes	µg/g
Tellurium:ICP:A	30	2	1	94.9400	yes	µg/g
Uranium:ICP:A	29	1	1	996.1200	yes	µg/g
Uranium:ICP:A	30	2	1	949.4000	yes	µg/g
Uranium:ICP:W	29	1	1	916.9000	yes	µg/g
Uranium-234:Mass Spectrometry:F	29	1	2	0.0030	no	%
Uranium-236:Mass Spectrometry:F	29	1	2	0.0061	no	%
Vanadium:ICP:A	29	1	1	9.9612	yes	µg/g
Vanadium:ICP:A	30	2	1	9.4940	yes	µg/g
Yttrium:ICP:A	29	1	1	9.9612	yes	µg/g
Yttrium:ICP:A	30	2	1	9.4940	yes	µg/g

A significant percentage of the results reported in Table 5-4 are ICP acid digestion or ICP water digestion methods. All of these results are below the detection limits and have a dilution factor of 10. Since these results are below the detection limits, the detection limits are used as the result values. The other results for these constituents and methods (i.e., those not listed in Table 5-4) have a dilution factor of 2, and are either close to or below the detection limit. The results with the dilution factor of 10 are roughly 5 times larger than those with a dilution factor of 2. These large differences (i.e., by a factor of 5) are due to the detection limit differences at the two dilution factors. These large detection limit differences are contributors to the substantial analytical variability in the cations subgroup noted earlier in this section (see Section 5.1).

The carbon-14 liquid scintillation result for direct sampling shown in Table 5-4 is from Sample 93-04316-J-1, according to the core 30 data report (Giamberardini 1993). This aliquot result (core 30, composite 1) is much higher than the other three results from the same core (not shown in Table 5-4), which show reasonable agreement with each other. The report notes that the relative percent difference (RPD) for the sample results is 133%, compared to 3.5% for the duplicate results. The report attributes the high RPD to the fact that the sample was nearly dry, which may cause inhomogeneity and difficulty in obtaining reliable analyses.

Two results from the water leach samples taken for the carbon-14 liquid scintillation analyses are also listed in Table 5-4. The core 30 data report (Giamberardini 1993) notes a wide discrepancy (by a factor of about 10) between sample and duplicate for both core 29, composite 2 and core 30, composite 2. The report offers no apparent reason for the anomalies.

The mercury by cold vapor atomic absorption (CVAA) result shown in Table 5-4 is one of two composite results noted in the core 30 data report (Giamberardini 1993). The report notes that the RPD for core 30, composite 2 is quite high (41%), indicating significant inhomogeneity for mercury within the composite. The RPDs for the other core/composite combinations are acceptable.

The remaining potentially anomalous results in Table 5-4 were not discussed in the core 29 and core 30 data reports (Giamberardini 1993). None of the results in Table 5-4 were excluded from any of the statistical analyses in this section.

5.2 PHYSICAL MEASUREMENTS

Measurements of such physical characteristics as shear strength, viscosity, particle size, and settling properties were taken. These measurements are necessary for the design and fabrication of retrieval, pretreatment, and final waste disposal systems. General physical assays were performed on samples from core 29. Particle size assays were performed on duplicate samples taken from the unhomogenized segments from both core 29 and core 30.

Sample rheology, which included shear strength and settling behavior, was run on the unhomogenized segments from core 29. Since holding time was exceeded, shear strength is a qualified estimate.

The physical measurements made on the waste are summarized in Table 5-5, which shows the averages of the available measurements (excluding those eliminated for the reasons cited above). A preferable set of measurements would include complete segment-level measurements on both cores, so that both horizontal and vertical variability could be adequately assessed.

Table 5-5. Summary of Core 29 Physical Measurements.

Analyte	Units	Segments	
		3	5
Segment - As Received			
Volume % settled solids	%	100	100
Density	g/mL	1.27	1.35
Volume % centrifuged solids	%	57	63
Weight % centrifuged solids	%	55	67
Centrifuged supernate density	g/mL	1.15	1.17
Centrifuged solids density	g/mL	1.38	1.45
Shear strength	dynes/cm^2	<300	900
Dissolved solids	%	11.6	9.6
Undissolved solids	%	18.6	27.6
Total solids	%	30.2	37.2
Segment - 1:1 Water to Sample Dilution			
Volume % settled solids	%	65.8	81.8
Density	g/mL	1.11	1.14
Segment - 3:1 Water to Sample Dilution			
Volume % settled solids	%	32.3	42.5
Density	g/mL	1.05	1.06

5.2.1 Physical and Rheological Properties

The important physical measurements recorded include density, temperature (in-situ), and three different measurements of weight percent solids. As indicated in Table 5-6, solids constitute roughly 36 to 37% (by weight) of the waste. The balance is presumed to be water.

Table 5-6. Weight Percent Solids.

Segment Level		
Segment	Core 29 Average wt%	Core 30 Average wt%
1	NO	NO
2	31.9	NO
3	31.9	33.0
4	35.2	35.1
5	36.3	31.5
Composite Level		
Composite		
1	36.3	37.5
2	36.3	37.9
Segment Level Average	33.8	33.5

NO = Not observed

The weight percent total solids analyses were performed on samples from the core composites. Weight percent solids was determined from duplicate samples according to technical procedure PNL-ALO-504. This analysis is a gravimetric determination of the weight percent solids as measured by the loss of mass in the sample after drying in an oven at 105 °C for 24 hours. The segment data was obtained on unhomogenized material in the High-Level Radioactive Facility, and the reported core composite data was obtained in the Shielded Analytical Laboratory on homogenized core composite material.

The weight percent total solids values for the core 29 composites were within experimental error, with an average value of 36.3 ± 0.1%. The average weight percent solids for core 30 composites was 37.7 ± 0.3%. These values compare well with the average of the segment level results, as seen in Table 5-6. The weight percent solids appear to be reasonably uniform between cores 29 and 30.

5.2.2 Shear Strength

The shear strength of the waste from tank B-111 was measured on the unhomogenized segment samples from core 29 (segments 3 and 5). The shear strength measurements were made at ambient temperature using a shear vane connected to a viscometer and rotated at 0.3 rpm, in accordance with technical procedure PNL-ALO-501. Shear strength is a semiquantitative measurement of the force required to displace the sample. Because shear strength is affected by sample handling, the measurement was taken without any sample homogenization.

The shear strengths measured were 900 dynes/cm^2 for segment 5, and < 300 dynes/cm^2 for segment 3. The shear stress of the material exceeded the baseline value for the measurement system (300 dynes/cm^2) in only one of the two cases. Because of the long lag time between sampling and analysis, these should be considered estimates.

5.2.3 Energetics

A summary of the thermal analysis is contained in Table 5-7. The most significant conclusion drawn from the thermal analysis is that no exotherms were found. Thermal measurements were made on all aliquots from unhomogenized segments of cores 29 and 30, so it is relatively certain that no exothermic layer exists in this waste.

Table 5-7. Cores 29 and 30 Thermal Measurements. (2 pages)

Transition	DSC			TGA	
	Enthalpy (cal/g)	Onset (°C)	Range (°C)	Range (°C)	Mass Loss (%)
Core 29 Segment 2					
1	289	97	30-150	30-130	57.5
2	1.2	175	167-193	125-500	2.3
3	3.0	209	192-230		
4	NO	NO	NO		
Core 29 Segment 3					
1	269	100	30-143	30-145	60.9
2	1.3	174	169-196	136-500	3.2
3	NO	NO	NO		
4	NO	NO	NO		
Core 29 Segment 4					
1	309	108	30-146	30-147	53.8
2	1.5	174	163-195	142-500	4.6
3	2.4	218	205-249		
4	3.6	317	298-356		
Core 29 Segment 5					
1	284	85	30-150	30-145	50.9
2	NO	NO	NO	145-500	5.5
3	1.6	222	211-265		
4	17.2	322	266-440		
Core 30 Segment 2					
1	287	72	30-144	30-155	63.4
2	0.3	179	173-196	137-500	3.8
3	2.0	216	195-249		
4	22.6	290	271-464		

Table 5-7. Cores 29 and 30 Thermal Measurements. (2 pages)

Transition	DSC			TGA	
	Enthalpy (cal/g)	Onset (°C)	Range (°C)	Range (°C)	Mass Loss (%)
Core 30 Segment 3					
1	285	81	30-150	30-140	61.8
2	0.5	177	172-199	137-500	3.8
3	1.6	224	206-262		
4	12.2	311	265-418		
Core 30 Segment 4					
1	270	94	30-165	30-160	54.7
2	NO	NO	NO	160-500	5.1
3	2.0	222	206-262		
4	26.0	312	260-458		
Core 30 Segment 5					
1	290	110	30-147	30-153	60.0
2	0.8	178	172-201	135-500	5.0
3	1.5	227	217-252		
4	20.3	310	253-416		

NO = Not observed

However, the thermal analysis did identify four endotherms in the waste, which absorbed approximately 300 cal/g in total. These endotherms occurred at approximately 94, 176, 219, and 310 °C, with most (95%) of the endothermic behavior occurring between ambient and 140 °C. The other endotherms are much smaller, and may represent either fluctuations associated with the baseline or stages in a series of endothermic events. Because of the relatively close proximity of Transitions 2 and 3 in temperature, their relatively small size, the qualitative nature of the assay, and the fact that no corresponding mass loss was observed during the thermogravimetric analysis (TGA), these endotherms are not considered fully credible. However, the endotherm observed with Transition 4 had a much more substantial signal in the differential scanning calorimetry (DSC). Therefore, this endotherm is considered credible, and potentially represents a physicochemical process occurring in the waste in that temperature range (277 to 500 °C).

5.2.4 Particle Size Analysis

Particle size distribution was measured on unhomogenized samples from each segment. The Brinkmann particle size analyzer, used in accordance with technical procedure PNL-ALO-530, Rev. 0, determines particle size in the range of 0.5 to 150 microns. Most of the particles in these samples were less than 20 microns in diameter. The median particle diameters, based on number and volume densities, are given in Table 5-8. The volume density data indicate that there is a small percentage of particles of much larger size, but it appears that only a few particles exceed 100 microns in diameter.

Table 5-8. Particle Size Distribution for Cores 29 and 30.

Segment	Particle size, microns (by number)			Particle size, microns (by volume)		
	Mean	Standard deviation	Median	Mean	Standard deviation	Median
Core 29						
2	1.23	1.46	8.96	28.74	16.49	30.91
3	1.46	1.55	8.96	13.61	16.88	9.89
4	1.31	1.39	8.91	21.18	28.58	11.58
5	1.53	1.51	1.16	11.12	6.11	10.62
Core 30						
2	21.58	23.37	9.62	21.58	23.37	9.62
3	1.23	1.16	8.89	11.89	9.66	7.67
4	8.94	8.43	8.85	6.62	7.46	2.57
5	1.15	8.95	8.92	22.78	19.36	16.40

5.2.5 pH Measurement

The pH of the water leaches of both core composite materials was measured according to technical procedure PNL-ALO-225. The average pH for the water leaches of the composites were 8.97 and 8.98 for composites 1 and 2 of core 29, and 8.79 and 8.74 for composites 1 and 2 of core 30, respectively.

5.3 HEAT LOAD ANALYSIS

The waste in tank B-111 is radioactive, and consequently generates some heat through radioactive decay. The most significant radioactive contributors in the waste are strontium-90 and cesium-137, contributing 264,000 and 168,000 curies, respectively. Table 5-9 summarizes the power produced by the radionuclides in the waste. About 2.5 kW of heat are produced in the tank, based on the heat load calculations--the equivalent of 25 ordinary 100-watt light bulbs. The heat load calculations indicate that there is modest heat production from the decay of the radioactive isotopes in the tank.

Table 5-9. Radionuclide Inventory and Project Heat Load.

	Total Ci	Watts/Ci	Watts
Americium-241	9.01 e+01	3.28e-02	2.96e+00
Cesium-137	1.68e+05	4.72e-03	7.96e+02
Cobalt-60	4.12e+00	1.54e-02	6.35e-02
Curium-242	9.79e-02	3.62e-02	3.54e-03
Curium-243/244	5.01e-01	3.44e-02	1.72e-02
Europium-154	1.81e+02	9.03e-03	1.63e+00
Europium-155	2.13e+02	7.27e-04	1.55e-01
Neptunium-237	7.61e-02	2.38e-02	1.81e-03
Plutonium-238	3.25e+00	3.33e-02	1.08e-01
Plutonium-239/240	1.04e+02	3.06e-02	3.18e+00
Strontium-90	2.64e+05	6.67e-03	1.76e+03
Technetium-99	1.21e+02	5.00e-04	6.06e-02
Thorium-232	3.24e-02	2.38e-02	7.72e-04
Tritium	2.93e+00	2.61e-01	7.63e-01
Total			2.57e+03

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6.0 INTERPRETATION OF ANALYTICAL RESULTS

This section contains a comparison of the analytical results from tank B-111 core samples with B-111 historical estimates, which are based on process knowledge. The tank B-111 analytical results are also compared to analytical results from tank B-110 core samples.

6.1 TANK WASTE PROFILE

As Table 5-1 shows, there are a limited number of segment-level analyses for tank B-111. All of the chemical analyses on the segment level are from segment homogenization tests. The three segments that were selected for the homogenization tests (segment 4 for core 29 and segments 3 and 5 for core 30) were not located appropriately to allow a tank profile analysis. Only a few physical properties measurements include complete segment data profiles for both cores. For these two reasons, no attempt was made to construct waste profiles from this small set of constituent data.

6.2 WASTE SUMMARY AND CONDITIONS

Table 6-1 compares historical data (Brevick 1994) to current sampling results. The second column in Table 6-1 presents the best predictions by LANL. These are the same results as those presented in Table 2-1. At present, the LANL estimates are considered the most authoritative historical estimates. Column 2 of Table 6-1 should be compared to column 3 (core sampling results) to determine the level of agreement between the LANL historical estimates and the core sampling results. For simplicity, only three significant digits are reported in columns 2 and 3 of Table 6-1.

The third and fourth columns of Table 6-1 list the mean concentration estimates and their associated RSDs, obtained from the ANOVA fits to the composite results, as described in Section 5.0. A complete tabulation of the mean concentration estimates for each constituent is shown in Appendix B. The final column in Table 6-1 presents the relative percent errors for the LANL predictions.

Of the 28 constituents and measurements listed in Table 6-1, 5 show relatively good agreement (i.e., relative percent error less than $\pm$ 50%) between the historical and composite data estimates. Included in these analytes are three of the major constituents mentioned in Section 5.0 (bismuth, iron, and sodium). Nitrite and phosphate are major constituents that exhibit poor agreement (i.e., relative percent error more than $\pm$ 100%) between the historical and composite data estimates.

Table 6-1. Comparison of Historical Versus Composite Concentration Estimates.

Analyte	LANL	Composite Data		Relative Percent Error*
	Est.	Est.	%-RSD	
	(μg/g)			
Aluminum	105	899	7	-88.28
Bismuth	21,900	20,200	1	8.21
Carbonate	211	NA	NA	NA
Chromium	544	1,110	5	-50.98
Fluoride	2,060	1,560	2	32.31
Hydroxide	25,700	NA	NA	NA
Iron	22,100	17,700	5	25.03
Lead	0.133	1,570	7	-99.99
Manganese	0	78.9	6	-100
Nitrate	36,400	82,000	8	-55.66
Nitrite	0	45,000	9	-100
Phosphate	97,900	48,700	3	101.03
Potassium	0	674	18	-100
Silicate	13,600	NA	NA	NA
Sodium	104,000	95,700	2	8.79
Sulfate	3,730	11,600	1	-67.82
Total organic carbon	152	875	12	-82.63
	(μCi/g)			
Americium-241	NA	0.0846	25	NA
Carbon-14	NA	0.00108	22	NA
Cesium-137	30.6	158	9	-80.65
Neptunium-237	NA	7.14e-05	22	NA
Plutonium	0.388	0.1	5	287.73
Plutonium-238	NA	0.00305	10	NA
Plutonium-239/240	NA	0.0973	5	NA
Strontium-90	1,040	248	22	319.25
Technetium-99	NA	0.114	10	NA
	(g/mL)			
Density	1.33	1.19	NA	11.76
	(%)			
Weight percent solids	34.5	37.0	2	-6.55

*Relative percent error: (Hist. Est. - Comp. Est)/(Comp. Est.) x 100.

From the comparison made in Table 6-1, it is concluded that the LANL estimates are generally within an order of magnitude of the sampling results for tank B-111, and provide an acceptable preliminary basis for waste tank inventory estimates. However, they are not substitutes for core sample data, should more detailed information be required.

Table 6-2 provides a means of determining internal consistency for the principal radionuclides. The gross alpha and gross beta measurements (from Table 5-2) are compared to the arithmetic mean of their respective main contributors (sum of alpha emitters = ^{241}Am + $^{239/240}$Pu; sum of beta emitters = $2(^{90}$Sr) + (^{137}Cs). The comparison shows very good agreement in both cases, with RPDs less than 5%.

Table 6-2. Alpha and Beta Energy Checks.

Calculation	Gross Alpha or Beta	RPD
Total Alpha		
^{241}Am + $^{239/240}$Pu = 0.182 μCi/g	0.176 μCi/g	3.4%
Total Beta		
$2(^{90}$Sr) + ^{137}Cs = 654 μCi/g	628 μCi/g	4.4%

6.3 COMPARISON OF B-110 AND B-111 SAMPLING RESULTS

The compositions of the waste in tanks B-110 and B-111 are expected to be somewhat similar. This is due to the fact that B-111 received waste via a cascade from tank B-110 for most of its service life (Anderson 1990; Agnew and Brown 1994). The LANL historical estimates, which are based on tank process history, are very similar for tanks B-111 and B-110.

This section contains a comparison between tanks B-111 and B-110 for a subset of the constituents (i.e., the major constituents (>0.5 wt%), plus total organic carbon (TOC), cesium-137, and strontium-90). This comparison is accomplished by fitting the following statistical model to composite data from both tanks:

$$Y_{ijk} = \mu + T_i + C_{ij} + E_{ijk} \tag{2}$$

where:

Y_{ijk} = The measured value of concentration of a constituent in core j of tank i

μ = The mean concentration over both tanks

T_i = The effect of tank i on the mean

C_{ij} = The effect of core j within tank i

E_{ijk} = The analytical error.

Table 6-3 shows the composite sample results for the two tanks. The results in the second and third columns are the B-110 means and corresponding RSDs taken from Heasler et al. (1993). The results in the fourth and fifth columns are the B-111 means and corresponding RSDs taken from Appendix B. The sixth column of Table 6-3 contains the p-values from the ANOVA, which tests whether or not the differences between the means are significant. This p-value is the probability that there is no difference between the tank means, given the observed sample results. If the p-value is less than 0.05, it is concluded that the tank means are significantly different from each other.

Table 6-3. Major Constituent Comparisons Between Tanks 241-B-110 and 241-B-111.

Constituent	B-110		B-111		p-value
	$\hat{\mu}$	% RSD $(\hat{\mu})$	$\hat{\mu}$	% RSD $(\hat{\mu})$	
	(μg/g)		(μg/g)		
Bismuth	1.85e+04	7	2.02e+04	1	0.472
Iron	1.81e+04	4	1.77e+04	5	0.740
Nitrate	1.87e+05	8	8.20e+04	8	0.001
Nitrite	1.03e+04	4	4.50e+04	9	0.001
Phosphate	2.53e+04	4	2.39e+04	3	0.436
Silicon	9.36e+03	4	1.04e+04	8	0.141
Sodium	9.77e+04	3	9.57e+04	2	0.805
Sulfate	1.15e+04	6	1.16e+04	1	0.688
Total Organic Carbon	3.81e+02	6	8.75e+02	12	0.885
	(μCi/g)		(μCi/g)		
Cesium-137	1.49e+01	4	1.58e+02	9	0.001
Strontium-90	1.08e+02	4	2.48e+02	22	0.045

There is reasonable agreement between most of the constituent means for tanks B-110 and B-111, with the exception of strontium-90, cesium-137, nitrate and nitrite. Cesium-137 and strontium-90, the two major radionuclides in both tanks, were found in greater concentration in B-111 than in B-110. From the Tank Layer Models for each tank, a contributing factor to the higher levels of cesium-137 and strontium-90 could be the amount of added PUREX waste (this is, specifically, P2 waste as defined by Brevick [1994]), a waste stream noted to be high in cesium-137 and strontium-90. These amounts of P2 are 7,600 L (2,000 gal) and 49,200 L (13,000 gal), respectively, for tanks B-110 and B-111 (Brevick 1994). Also, the fact that the ratio of nitrate to nitrite is much smaller for tank B-111 than for B-110 could be caused by the radiolytic conversion of nitrate to nitrite occurring in both tanks. This process is accelerated by the presence of higher levels of cesium-137 and strontium-90. No statistical tests were conducted to determine whether the estimates of uncertainty were similar between tanks B-111 and B-110. However, the RSDs of the means give some indication that the uncertainties are similar.

This brief comparison between the sample results from tanks B-110 and B-111 adds strength to the argument that waste from these two tanks can be treated similarly. However, a more detailed analysis should be carried out on all of the constituents measured in both of these tanks, to make the comparison more complete.

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7.0 QUANTITATIVE AND STATISTICAL QA TESTS

This section contains a summary of the various QA tests and measurements applied to the tank B-111 analytical results. These tests and measurements include the mass and charge balance, homogenization tests, spike recoveries, and method blanks.

7.1 MASS AND CHARGE BALANCE

The mass and charge balance is a validation calculation, designed to compare the results of the metals, anions, and moisture laboratory measurements for consistency with each other. The best estimates of tank contents for the metals and anions are summed in order to postulate the amount of water present in the tank. The postulated water content is compared to the measured water content for agreement.

Since two substantial analyte measurements were not made, oxygen and complexed hydroxide, assumptions are made to account for them. For oxygen, it is assumed that all the boron, phosphorus, selenium, silicon, and tellurium measured in the core samples are present in their oxygenated anion forms, as shown in the fifth column of Table 7-1. To determine complexed hydroxide, a charge balance is calculated, and the appropriate amount of hydroxide is added to balance the charges.

Table 7-1. Anion Mass and Charge Balance Contribution with Postulated Oxy-Anions.

| Anion | Mass | | Charge | Postulated Oxygen | | |
	μg/g	RSD	μmol/g	Anion	μg/g	RSD
Boron	51	7	2.38	$B_4O_7^{-2}$	133	7
Chloride	1,020	2	28.77			
Cyanide	2	19	0.07			
Fluoride	1,560	2	82.11			
Nitrate	82,000	8	1322.58			
Nitrite	45,000	9	987.26			
Phosphorus	15,900	8	1540.2	PO_4^{-3}	32,913	8
Selenium	22	15	1.12	SeO_3^{-2}	13	15
Silicon	10,400	8	742.86	SiO_3^{-2}	17,784	8
Sulfate	11,600	1	362.5			
Tellurium	21	5	0.32	TeO_3^{-2}	8	6

Table 7-1 lists the anions with postulated oxy-anions used in the mass and charge balances, while Table 7-2 lists the metals (cations). Table 7-3 shows the solubility of the phosphorus (as phosphate) by comparing the water-soluble portion to the total phosphate. The phosphate for this waste matrix is 47 to 49% soluble. All the concentrations listed in both tables are the best estimates of tank contents, taken from Appendix B. These tables also list the RSD associated with each estimate and its postulated charge. The RSDs are used to calculate the uncertainties associated with the mass totals.

Table 7-2. Metals (Cations) Mass and Charge Contribution.

Metal	Mass		Charge	Metal	Mass		Charge
	µg/g	RSD	µmol/g		µg/g	RSD	µmol/g
Aluminum	899	7	99.96	Antimony	11	9	0.26
Arsenic	28		1.12	Barium	28	11	0.41
Beryllium	2		0.39	Bismuth	20,200	1	289.98
Cadmium	3	15	0.05	Calcium	689	23	34.38
Cerium	21	9	0.44	Chromium	1,110	5	64.04
Cobalt	4	21	0.15	Copper	201	94	6.33
Dysprosium	7		0.13	Europium	3		0.07
Gadolinium	70		1.33	Iron	17,700	5	950.81
Lanthanum	7	6	0.15	Lead	1,570	7	15.16
Lithium	7		1.00	Magnesium	195	2	16.04
Manganese	79	6	2.87	Molybdenum	42	9	2.61
Neodymium	22	23	0.46	Nickel	19	3	0.63
Palladium	52		0.99	Potassium	674	18	17.24
Rhodium	35		1.02	Ruthenium	17		0.52
Sodium	95,700	2	4162.72	Strontium	218	2	4.98
Thallium	174		0.85	Thorium-232	279		4.81
Tin	279		9.40	Titanium	8	14	0.66
Tungsten	28		0.91	Uranium	197	4	4.97
Vanadium	2	12	0.24	Yttrium	2	21	0.08
Zinc	111	50	3.40	Zirconium	14	29	0.63

Table 7-3. Phosphate Solubility.

Calculation	Phosphate Solubility
$\dfrac{IC:W\ PO_4^{3-}\ result}{ICP:F\ P\ result\ as\ PO_4^{3-}} = \dfrac{23,900}{58,700}\ x\ 100$	49.1%
$\dfrac{IC:W\ PO\ result}{ICP:F\ P\ result} = \dfrac{7,520}{15,900}\ x\ 100$	47.3%

Table 7-4 summarizes the mass and charge balances from Tables 7-1 and 7-2, along with uncertainties associated with each total (expressed as RSD). Total charges are listed again in the fourth column, and from these totals the excess negative charge is determined. This excess negative charge is assigned to hydroxide, and the charge balance determines the mass of hydroxide in Table 7-4. The mass concentration, $\mu g/g$, or parts per million, resulting from the cations, anions, and predicted hydroxide is therefore subtracted from 1 million to estimate the water content. From Table 7-4, the postulated water content in the waste is 63.7%, within 1% agreement with the measured result. The estimated total mass is 994,000 $\mu g/g$ which is only -0.6% different from the total mass (1,000,000 $\mu g/g$) of the waste. As one can see from this mass balance, the assumptions made concerning the hydroxide and oxygen seem to fit the data well.

Table 7-4. Summary of Mass/Charge Balance.

Source	Mass		Charge
	$\mu g/g$	RSD	$\mu mol/g$
Sum of Cations (Metals)	140,708	2	5,702
Sum of Anions	167,576	4	-2,777
Estimated Oxygen	50,851	6	-2,284
Estimated Hydroxide	3,990	0	-641
Subtotal	363,000	NA	0
Postulated H_2O from Mass Balance	637,000	1	
Measured H_2O	630,000	2	
Relative Percent Difference (H_2O)	1%		
Estimated Total (subtotal + H_2O)	994,000		
Percent Difference from Total	-0.6%		

7.2 HOMOGENIZATION TESTS

Homogenization is a very important step in the process of making representative core composite samples. There were two homogenization steps for core samples from B-111. First, the segments from each core were homogenized. Then, samples were taken from the top and bottom of segment 4 from core 29 and segments 3 and 5 from core 30. Finally, homogenized waste from each segment was homogenized into composite samples of each core. The samples were prepared by potassium hydroxide (KOH) fusion and chemically analyzed using ICP and GEA to determine whether the sample homogenization was adequate.

The analytical results from the top and bottom segment samples (homogenization samples) were fit to the following nested random effects model:

$$Y_{ijk} = \mu + C_i + S_{ij} + H_{ijk} + E_{ijkl} \tag{3}$$

where:

Y_{ijk} = The measured value of concentration of a constituent in segment j of core i

μ = The mean concentration of the constituent

C_i = The core sampled

S_{ij} = The segment from the core

H_{ijk} = The location on the segment (homogenization effect)

E_{ijkl} = The analytical error.

The objective of the homogenization test is to determine whether the variability in the results between sampling locations is greater than zero. This objective can be met by analyzing the results of an ANOVA on the random effects model.

The results of the ANOVA are presented in Table 7-5. The homogenization RSD (estimated variability between locations relative to the mean) is given, together with the p-value from the homogenization tests. Each p-value listed in the table is the probability of obtaining the tabulated RSD value, given that the homogenization variability (σ_H^2) is really equal to zero. If the p-value is less than 0.01, it is concluded that σ_H^2 is greater than 0 (at the 99% confidence level). Analytes with more than 75% of the analytical results below the detection limits were excluded from this analysis.

Table 7-5. Homogenization Test Results.

Segment Level Homogenization Tests (Acid Digestion ICP and GEA)								
Analyte	Homogenization		<DL	Obs.	Analyte	Homogenization		<DL	Obs.
	RSD (%)	p-value				RSD (%)	p-value		
Aluminum	9	0.141	0	12	Barium	0	0.487	4	12
Bismuth	4	0.334	0	12	Boron	0	0.531	1	12
Cadmium	4	0.354	0	12	Calcium	13	0.019	0	12
Chromium	8	0.097	0	12	Copper	29	0.004	3	12
Iron	7	0.138	0	12	Lead	15	0.021	0	12
Magnesium	9	0.157	4	12	Manganese	9	0.017	0	12
Phosphorus	8	0.085	0	12	Silicon	13	0.016	0	12
Silver	0	0.539	5	12	Sodium	10	0.055	0	12
Strontium	7	0.110	0	12	Titanium	4	0.353	4	12
Zinc	15	0.002	4	12	Americium-241	0	0.790	7	12
Cesium-137	1	0.023	0	12	Europium-154	0	0.673	4	12
Europium-155	13	0.007	4	12	Gross alpha	6	0.281	0	12

The homogenization tests on the segment data show that for 88% of the analytes tested, the variability due to homogenization cannot be distinguished from zero (99% significance level). For the other 12% of the analytes (zinc, europium-155, and copper), the homogenization RSDs are relatively small (i.e., 10% to 15%), with the exception of copper. In general, the segment homogenization is considered adequate for B-111.

7.3 EVALUATION OF SPIKES AND BLANKS

Spikes and blanks are regularly run in the laboratory to determine whether or not the analysis procedures are producing unbiased measurements. If the results for the blanks are too high, or if the spike recoveries deviate substantially from 100%, then the associated measurements are either re-run or flagged in the database. The control thresholds used in this QA evaluation have been borrowed from the ground water standards contained in the *Resource Conservation and Recovery Act of 1976* (RCRA), and are not necessarily the most relevant standards to apply to these measurements.

In this section, we present an overview of the blank and spike measurements. These measurements provide a good indication of laboratory performance, but we have not attempted to apply the RCRA standards rigorously to this data. For the analysis presented in other parts of this report, all data, including QA flagged data, has been used. There was also no attempt to correct any of the data for high blanks or low spike recovery.

7.3.1 Quality Assurance Flags

Hanford Analytical Services (HAS) reviewed all data and assigned QA flags to the results. Of the 4,625 measurements in the data set, HAS classified about 12% as unusable or "estimate only" (a QA flag of J or Q). All these measurements were used in the analyses. About 49% of the measurements were below the detection limit (i.e., the analyte was not found in the samples).

In order to perform the analysis presented in this report, all data were used and none of the HAS-flagged data were deleted. Table 7-6 provides a list of the defined HAS flags, while Table 7-7 summarizes the amount of flagged data in the data set. From the tables, one can see that much of the data has been flagged as below detection limit (U and UJ); this is not a QA problem. The "Q" flag in Table 7-7 indicates that the result is close to the detection limit (i.e., above the detection limit but below the quantification limit).

Table 7-6. Quality Assurance Flag Description.

Flag	Meaning
B	Indicates compound was found in the blank.
C	Concerns not requiring qualification of the data but still having a potential impact on data quality.
E	Indicates that measurement was outside of the calibration range.
J	Indicates an estimated value for target and tentatively identified compounds; spectra meet criteria, but response is below Contract Required Quantitation Limit for the target compounds.
N	Material was not analyzed for, since the sample preparation made such measurement not appropriate (e.g., potassium in KOH/NI fusion preparation).
O	Measurement was beyond the range of the instrument.
Q	Associated results are qualitative.
R	Data are unusable.
S	Minimum detection limit was substituted for the reported value of the analytical result.
U	Indicates the compound was analyzed for, but not detected. The U-flagged concentration is the Contract Required Quantitation Limit.
X	Indicates compound was manually deleted because all requirements were not met.

Table 7-7. Summary of Quality Assurance Flags on Sample and Duplicate Measurements.

Analysis Method	NF	J	Q	U	UJ
AA (As):A	0	0	0	4	4
AA (Sb):A	0	0	0	4	4
AA (Se):A	0	0	0	4	4
CVAA (Hg):A	0	0	0	0	0
ICP:A	186	0	96	178	0
CVAA (Hg):A	4	4	0	0	0
DSC:D	228	0	0	0	0
Extractable Organic Halides	0	0	0	0	8
Extraction Organic (SVOA)	55	9	0	511	0
Alpha Radiochemistry:F	74	0	0	0	0
Beta Radiochemistry:F	24	0	0	0	0
GEA:F	65	0	0	23	0
ICP:F	246	0	134	500	0
Laser Fluorimetry:F	8	0	0	0	0
Liquid Scintillation:F	8	0	0	0	0
Mass Spectroscopy:F	32	0	0	0	0
Liquid Scintillation:W	10	0	0	6	0
Liquid Scintillation:A	8	0	0	0	0
Percent Solids:D	10	11	0	0	0
Persulfate Oxidation (TOC):D	12	12	0	0	0
Physical Properties	19	30	0	1	0
TGA:D	96	0	0	0	0
CN:W	3	4	0	1	0
Calorimetric:W	4	4	0	0	0
ICP:W	70	0	43	301	0
IC:W	24	24	0	0	0
ISE (NH3):W	4	4	0	0	0
TIC, TOC, TC:W	12	12	0	0	0
PH:W	4	0	0	0	0
Total Flags	1,206	114	273	1,533	20

NF = No flags

From Table 7-7, one can see that approximately one-third of all ICP-Fusion and ICP-Acid measurements above the detection limit have a Q flag. Since ICP is the major measurement method for a substantial number of analytes, there would be a large problem with data interpretation if all Q-flagged measurements were deleted from the ANOVA.

7.3.2 Blanks

To evaluate blanks, the ratio between the blank measurement and the average of the sample and its duplicate was computed. Since this ratio would have little meaning when the measurement is at or below the detection limit, any measurements at or below detection limits were eliminated. Also, a substantial number of measurements were eliminated because they did not have an associated sample identification number. Approximately 25% of the blanks in the data base had no sample identification numbers.

Table 7-8 presents a summary of the blank/measurement data. The table presents the median and maximum ratios for each measurement method, along with the 75% quantile. The distribution of the blank/measurement ratios is also presented graphically in Figure 7-1.

Table 7-8. Summary of Blank Analyses for Measurements Above Detection Limit.

Method	Below DL	Above DL	Median	75-quantile	Maximum
ICP:A	178	282	14	55	200
CVAA (Hg):A	0	8	1	1	1
Extraction Organic (SVOA)	511	64	200	200	200
Alpha Radiochemistry:F	0	74	0	0	51
Beta Radiochemistry:F	0	24	0	1	1
GEA:F	23	65	0	0	1
ICP:F	500	380	36	67	200
Laser Fluorimetry:F	0	8	0	0	0
Liquid Scintillation:F	0	8	37	45	53
Liquid Scintillation:W	6	10	15	16	17
CN-	1	7	45	50	56
Calorimetry:W	0	8	62	67	71
ICP:W	301	113	22	84	115
IC:W	0	48	0	3	4
ISE (NH3):W	0	8	15	19	23
TIC, TOC, TC:W	0	24	3	9	12

Figure 7-1. Blank/Measurement Ratios for Measurements Above Detection Limit.
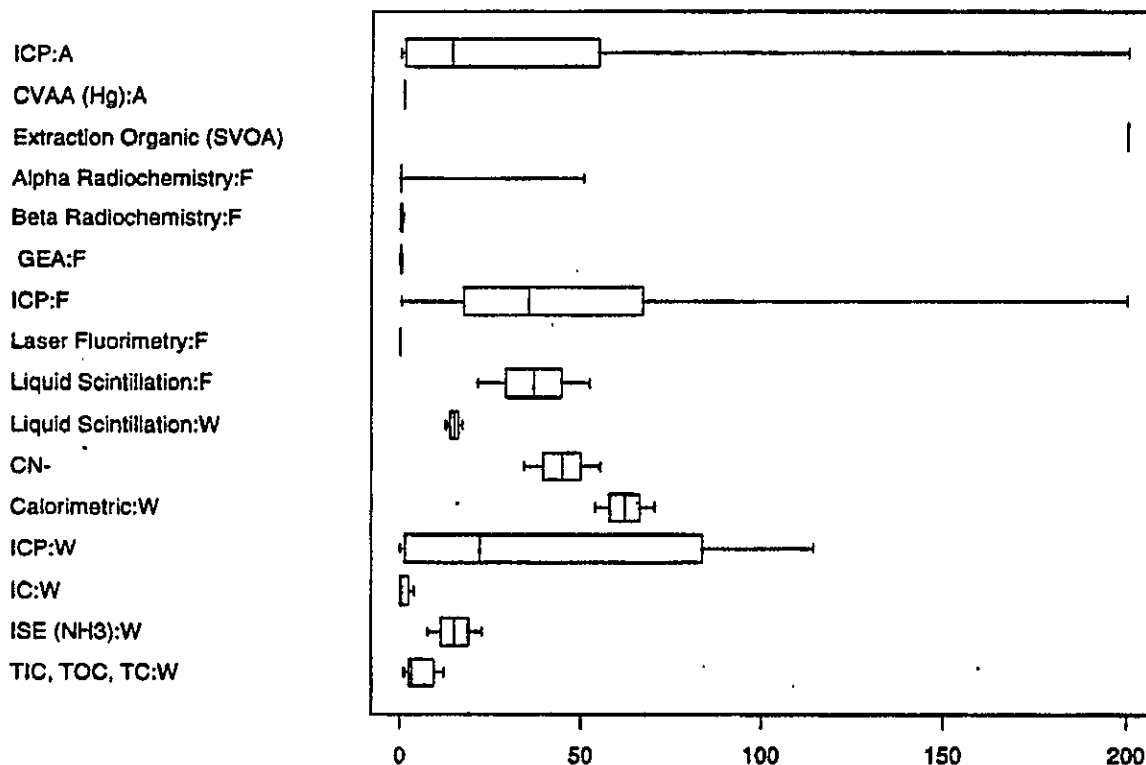
As can be seen from Figure 7-1, many of the blanks are high. Some measurement methods show very small blank/measurement ratios (such as CVAA, radiochemistry, GEA, and laser fluorimetry). On the other hand, ICP, the major measurement method, shows a fairly large blank effect; for acid digestion the median blank/measurement ratio is 14%, for water digestion the median ratio is 22%, and for KOH fusion it is a very substantial 36%. These results are not surprising because ICP measurement methods are commonly known to have large blank/measurement ratios. A common laboratory practice is to use the blank measurements to correct for background effects, and these measurements provide evidence that alterations in laboratory procedure may be appropriate.

Table 7-9 presents 10 of the analytes with the highest blank/measurement ratios. Many of these blanks are small in absolute terms (a few ppm) and close to the detection limit, so a large relative bias should not be too important. Even though two constituents listed in Table 7-9 (boron and uranium) are substantially above their detection limits and also exhibit large blank/measurement ratios, their overall concentrations in the waste are not high enough to warrant further action. It is interesting to note that one of the boron duplicate measurements is not flagged, even though it is substantially less than the blank.

Table 7-9. Examples of the Worst Blank Measurements.

Sample Id	Analyte	Analytical Method: Sample Preparation	Result (µg/g)	Result Type	Flags
93-04312a1	Cadmium	ICP:A	2.000	DUPLICATE	Q
93-04312a1	Cadmium	ICP:A	5.000	BLANK	
93-04312a1	Cadmium	ICP:A	2.000	PRIMARY	Q
93-4316h1	Boron	ICP:F	186.000	DUPLICATE	
93-4316h1	Boron	ICP:F	568.000	BLANK	
93-4316h1	Boron	ICP:F	66.000	PRIMARY	Q
93-04313-E1	Bis(2-ethylhexyl)phthalate	SVOA	2.800	DUPLICATE	J
93-04313-E1	Bis(2-ethylhexyl)phthalate	SVOA	10.000	BLANK	U
93-04313-E1	Bis(2-ethylhexyl)phthalate	SVOA	3.000	PRIMARY	J
93-04312-E1	Bis(2-ethylhexyl)phthalate	SVOA	3.100	DUPLICATE	J
93-04312-E1	Bis(2-ethylhexyl)phthalate	SVOA	10.000	BLANK	U
93-04312-E1	Bis(2-ethylhexyl)phthalate	SVOA	2.900	PRIMARY	J
93-4316h1	Barium	ICP:F	53.000	DUPLICATE	Q
93-4316h1	Barium	ICP:F	80.000	BLANK	
93-4316h1	Barium	ICP:F	37.000	PRIMARY	Q
92-04062H-1T	Cadmium	ICP:F	16.000	DUPLICATE	Q
92-04062H-1T	Cadmium	ICP:F	24.000	BLANK	Q
92-04062H-1T	Cadmium	ICP:F	21.000	PRIMARY	Q
93-4316h1	Cadmium	ICP:F	14.000	DUPLICATE	Q
93-4316h1	Cadmium	ICP:F	18.000	BLANK	Q
93-4316h1	Cadmium	ICP:F	14.000	PRIMARY	Q
93-4316h1	Silver	ICP:F	72.000	DUPLICATE	
93-4316h1	Silver	ICP:F	87.000	BLANK	
93-4316h1	Silver	ICP:F	76.000	PRIMARY	
93-4316c1	Calcium	ICP:W	7.000	DUPLICATE	Q
93-4316c1	Calcium	ICP:W	8.020	BLANK	U
93-4316c1	Calcium	ICP:W	5.100	BLANK	Q
93-4316c1	Calcium	ICP:W	5.000	PRIMARY	Q
93-4316h1	Cobalt	ICP:F	21.000	DUPLICATE	Q
93-4316h1	Cobalt	ICP:F	21.000	BLANK	Q
91-10553H-1T	Cadmium	ICP:F	34.000	DUPLICATE	Q
91-10553H-1T	Cadmium	ICP:F	31.000	BLANK	Q
91-10553H-1T	Cadmium	ICP:F	28.000	PRIMARY	Q
93-04316a1	Yttrium	ICP:A	2.000	DUPLICATE	Q
93-04316a1	Yttrium	ICP:A	1.972	BLANK	U
93-04312a1	Vanadium	ICP:A	2.000	DUPLICATE	Q
93-04312a1	Vanadium	ICP:A	1.958	BLANK	U
93-04312a1	Vanadium	ICP:A	2.000	PRIMARY	Q
93-04312a1	Silver	ICP:A	2.000	DUPLICATE	Q
93-04312a1	Silver	ICP:A	1.958	BLANK	U
93-04312a1	Silver	ICP:A	2.000	PRIMARY	Q
93-4312c1	Uranium	ICP:W	196.000	DUPLICATE	Q
93-4312c1	Uranium	ICP:W	188.900	BLANK	U
93-4312c1	Calcium	ICP:W	14.000	DUPLICATE	Q
93-4312c1	Calcium	ICP:W	16.200	BLANK	Q
93-4312c1	Calcium	ICP:W	20.000	PRIMARY	Q
93-4312h1	Boron	ICP:F	82.000	DUPLICATE	Q
93-4312h1	Boron	ICP:F	70.000	BLANK	Q
93-4312h1	Boron	ICP:F	65.000	PRIMARY	Q

7.3.3 Spikes

Spike recovery percentages are generally between 75% and 125%, except for the selenium and CN- measurements. Figure 7-2 and Table 7-10 provide concise summaries of the percent recoveries. As can be seen from Table 7-10, only 6 spikes are outside the range, and they are listed in Table 7-11.

Even though most of the recoveries are within the desired 75-125%, one should consider whether this information should be used to correct for biases. For several important measurement methods (i.e., fusion GEA, alpha and beta radiochemistry), the results are consistently above or below 100% recovery (see Figure 7-2). This consistency in the recoveries indicates that a bias may exist in these measurements. The variability in the recovery percentages is surprisingly small for several analysis methods.

Figure 7-2. Boxplots of Recovery Percentages.
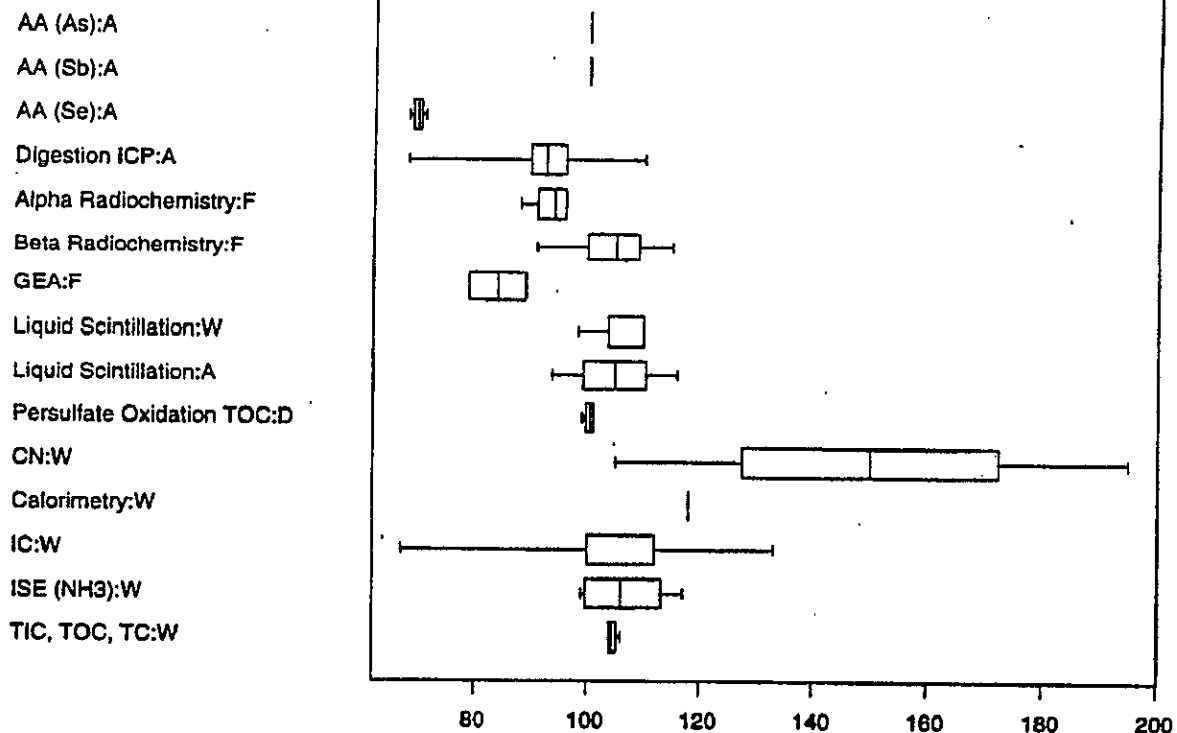
Table 7-10.   Summary of Spike Recoveries (75-125% Range).

Analytical Method	Outside	Inside
AA (As):A	0	1
AA (Sb):A	0	1
AA (Se):A	2	0
CVAA (Hg):A	0	0
ICP:A	1	0
CVAA (Hg):A	0	0
DSC:D	0	0
Extractable Organic Halides	0	0
Extraction Organic (SVOA)	0	0
Alpha Radiochemistry:F	0	24
Beta Radiochemistry:F	0	16
GEA:F	0	8
ICP:F	0	0
Laser Fluorimetry:F	0	0
Liquid Scintillation:F	0	0
Mass Spectroscopy:F	0	0
Liquid Scintillation:W	0	6
ICP	0	0
Liquid Scintillation:A	0	2
Percent Solids:D	0	0
Persulfate Oxidation (TOC):D	0	4
Physical Properties	0	0
TGA:D	0	0
CN:W	1	1
Calorimetric:W	0	1
ICP:W	0	0
IC:W	2	7
ISE (NH3):W	0	4
TIC, TOC, TC:W	0	4
pH:W	0	0

Table 7-11. Spike Recoveries Below 75% and Above 125%.

Sample ID	Method Name	Analyte	Result	Result Type	Flags
93-04316-C	IC:W	Chloride	67%	SPIKE-RECOVERY	
93-04316a1	ICP:A	Silicon	68%	SPIKE-RECOVERY	
93-04316-B	AA (Se):A	Selenium	68%	SPIKE-RECOVERY	
93-04312-B	AA (Se):A	Selenium	71%	SPIKE-RECOVERY	
93-04316-C	IC:W	Fluoride	134%	SPIKE-RECOVERY	
93-04313-C	CN:W	Cyanide	195%	SPIKE-RECOVERY	

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8.0 CONCLUSIONS AND RECOMMENDATIONS

The waste in tank B-111 is made up primarily of 2C waste from the bismuth phosphate process and FP waste. The 2C waste is expected to have relatively low radioactivity levels and is expected to be found in the bottom portion of tank B-111. The FP waste has higher levels of radioactivity (strontium-90 and cesium-137) and is expected to be found on top of the 2C waste. The sampling data could not be used to verify that these distinct waste layers exist, since very few segment level analyses were performed for tank B-111.

The analytes found in highest concentration ($> 10^4$ ppm) for the B-111 samples in descending order are water, sodium, nitrate, phosphate, nitrite, bismuth, iron, sulfate, and silicon.

The uncertainties in the best estimates (see Appendix B) produced in this study (from composite data) are generally dominated by horizontal spatial variability. This characteristic has consequences for tank sampling. If more accurate estimates of the tank contents are required, then more core samples must be taken (improvements in analytic procedures or in sampling methodology would not be adequate).

The tank B-111 sampling results were compared to the LANL historical estimates for B-111 and to B-110 sampling results (B-110 and B-111 have similar process histories). The LANL estimates are generally within an order of magnitude of the sampling results. More specific comparisons and conclusions cannot be made since the uncertainty in the LANL estimates cannot be quantified. There is good agreement between the sampling results for tanks B-111 and B-110 for six out of eight major constituents. This comparison suggests that the waste in these two tanks can be treated similarly; however, a much more detailed comparison should be made to see if there is agreement over all the constituents measured in these tanks. A comparison of the uncertainty observed in each tank would also be in order.

The QA tests show mixed results as to the usability of the analytical data from B-111 core samples. The mass/charge balance shows good agreement between postulated and measured results. In general, homogenization tests indicate that the waste samples from B-111 were mixed sufficiently to produce representative results. The analysis of spikes and blanks, however, reveals some problems with the data. The majority of the spike recoveries are within the 100% ± 25% acceptable range; however, some analytical methods had spike recoveries that were consistently above or below 100%. This consistency in recoveries indicates that a bias may exist in the sampling. This is a problem that should be addressed. It was also noted in Section 7.3.2 that the blank/measurement ratios for the ICP methods were quite high (i.e., 14% to 36%), and that alterations in laboratory procedure to correct for this bias may be warranted. However, the analytes whose concentrations are relatively large do not appear to demonstrate the bias observed in the lower concentration analytes. There were no attempts to use these blank measurements to correct any of the results due to lack of sufficient data regarding the process performance of the analytical laboratories.

Hanford Analytical Services reviewed the B-111 core reports and flagged 12% of the data as unusable and 5% more as suspect. It was noted that the validation criteria used (groundwater) may not be appropriate for the sample matrices. In order to perform all of the analyses in this report, all data was used and none of the HAS-flagged data was deleted. More applicable criteria should be sought or developed to account for the relatively unique characteristics and hazards associated with mixed wastes.

B-111 is not on any of the watch lists (e.g., ferrocyanide or flammable gas), and therefore has no safety issues that need to be addressed.

9.0 REFERENCES

Agnew, S. F., and T. M. Brown, 1994, *Waste Status and Transaction Record Summary For the Northeast Quadrant*, WHC-SD-WM-TI-615, Westinghouse Hanford Company, Richland, Washington.

Anderson, J. D., 1990, *A History of the 200 Area Tank Farms*, WHC-MR-0132, Westinghouse Hanford Company, Richland, Washington.

Brevick, C. H., 1994, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Areas*, WHC-SD-WM-ER-349, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

De Lorenzo, D. S., J. H. Rutherford, D. J. Smith, D. B. Hiller, K. W. Johnson, and B. C. Simpson, 1994, *Tank Characterization Reference Guide*, WHC-SD-WM-TI-648, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

Giamberardini, K. K., 1993, *PNL 325 Laboratories Single Shell Tank Waste Characterization, Tank B-111 Cores 29 and 30*, WHC-SD-WM-DP-041, Westinghouse Hanford Company, Richland, Washington.

Ecology, EPA, and DOE, 1994, *Hanford Federal Facility Agreement and Consent Order*, as amended, Washington State Department of Ecology, U.S. Environmental Protection Agency, and U.S. Department of Energy, Olympia, Washington.

Hanlon, B. M., 1993, *Tank Farm Surveillance and Waste Status Summary Report for November 1993*, WHC-EP-0182-68, Westinghouse Hanford Company, Richland, Washington.

Heasler, P. G., C. M. Anderson, D. B. Baird, R. J. Serne, and P. D. Whitney, 1993, *Statistical Evaluation of Core Samples From Hanford Tank B110*, PNL-8745, Pacific Northwest National Laboratory, Richland, Washington.

Jungfleisch, F. M., and B. C. Simpson, 1993, *A Preliminary Estimation of the Waste Inventories in Hanford Tanks Through 1980*, WHC-SD-WM-TI-057, Rev. O-A, Westinghouse Hanford Company, Richland, Washington.

Keller, C. M., 1993, *Core Drill Operating Envelope Test Report*, WHC-SD-WM-ER-123, Westinghouse Hanford Company, Richland, Washington.

Resource Conservation and Recovery Act of 1976, 42 USC 6901, et seq.

Technical Procedure PNL-ALO-225, 1990, "Measurement of pH in Aqueous Solutions," Pacific Northwest National Laboratory, Richland, Washington.

Technical Procedure PNL-ALO-501, "Physical Rheological Properties," Pacific Northwest National Laboratory, Richland, Washington.

Technical Procedure PNL-ALO-504, "Percent Solids Determination of Soils/Sludges/Solids," Pacific Northwest National Laboratory, Richland, Washington.

Technical Procedure PNL-ALO-530, Rev. 0, 1989, "Particle Size Distribution By Laser Scanning (Time of Transition)," Pacific Northwest National Laboratory, Richland, Washington.

WAC 173-303, "Dangerous Waste Regulations," *Washington Administrative Code*, as amended.

10.0 BIBLIOGRAPHY

Babad, H., 1994, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Westinghouse Hanford Company, Richland, Washington.

Bell, K. E., 1993, *TWRS Tank Waste Characterization Plan*, WHC-SD-WM-PLN-047, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

Hanford Works Monthly Report, February 1948, HW-9191 DEL.

Hanford Works Monthly Report, March 1948, HW-9595 DEL.

Huckaby, J. L, 1992, *Characterization of Vapors in Single-Shell Tanks Scheduled for Rotary-Mode Sampling*, WHC-SD-WM-TI-536, Westinghouse Hanford Company, Richland, Washington.

Winters, W. I., L. Jensen, L. M. Sasaki, R. L. Weiss, J. F. Keller, A. J. Schmidt, and M. G. Woodruff, 1990, *Waste Characterization Plan for Hanford Site Single-Shell Tanks*, WHC-EP-0210, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

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APPENDIX A

TANK ENGINEERING DATA AND WASTE SUMMARY

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Figure A-1. Top View of Tank 241-B-111.
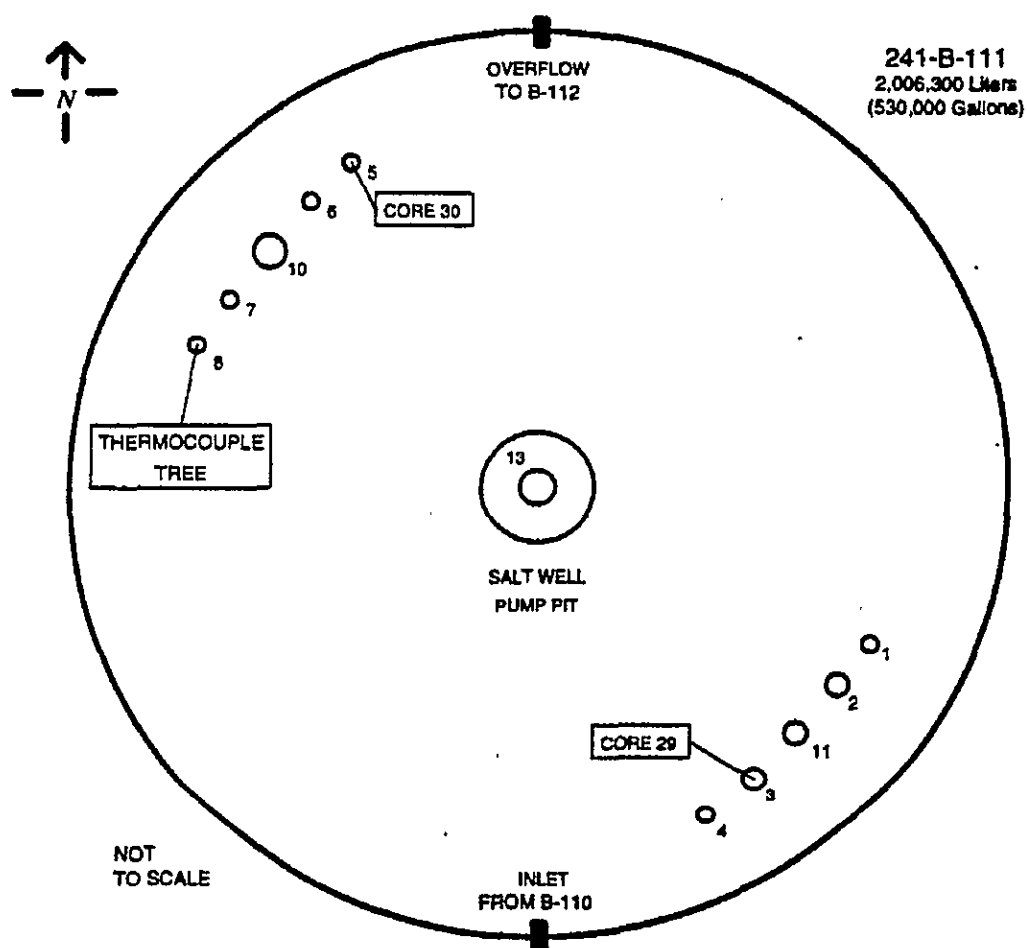
Table A-1. Engineering Data Summary of Tank 241-B-111.

Tank Engineering Description	
Type:	Single-Shell Tank
Construction:	1943-1944
In-Service:	December 1945
Out of Service:	April 1976
Diameter:	23 m (75 ft)
Operating Depth:	5.2 m (17 ft)
Nominal Capacity:	2,006,300 L (530,000 gal)
Bottom Shape:	Dish
Hanford Coordinates:	N45337.5, W52852.5
Ventilation:	Passive
Tank Status	
Watch List:	None
Interim Stabilized:	June 1985
Interim Isolated:	October 1985
Contents:	Non-Complex Waste
Integrity Category:	Assumed Leaker (1978) (30,300 L [8,000 gal])

Table A-2. Inventory Summary of Tank 241-B-111.

Physical Properties of Waste:			
Total Waste:	897,100 L (237,000 gal)	Supernate Volume:	3,800 L (1,000 gal)
Drainable Inter. Liquid:	79,500 L (21,000 gal)	Density:	1.190 g/mL
H_2O Average:	63.1%	Total Waste Mass:	1,067,600 kg
pH:	8.87	Temperature Average:	26.7 °C (80.2 °F)
Heat Load:	2.57e+03 watts	Maximum Exotherm:	No Exotherms
Chemical Properties of Waste			
Sodium:	1.02e+05 kg (9.57 wt%)	Bismuth:	2.15e+04 kg (2.02 wt%)
Nitrate:	8.74e+04 kg (8.20 wt%)	Iron:	1.89e+04 kg (1.77 wt%)
Phosphate:	5.18e+04 kg (4.87 wt%)	Sulfate:	1.24 e+04 kg (1.16 wt%)
Nitrite:	4.79e+04 kg (4.50 wt%)	Silicon:	1.11e+04 kg (1.04 wt%)
Radionuclides in the Waste			
Total Alpha Pu:*	1.07e+02 Ci	Strontium-90:	2.64e+05 Ci
Cesium-137:	1.68e+05 Ci	Total Uranium:	2.10e+02 kg (0.02 wt%)

*Total alpha emitted from ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu.

APPENDIX B

COMPOSITE ESTIMATES AND VARIABILITY SUMMARY

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APPENDIX B

COMPOSITE ESTIMATES AND VARIABILITY SUMMARY

This section tabulates analysis of variance results for the composite data (including the drainable liquid). The most important value in this table is the average concentration estimate for each constituent, $\hat{\mu}$, but the table also presents variance component estimates. The model used to produce these results is:

$$Y_{ijk} = \mu + C_i + S_{ij} + E_{ijk} \qquad (4)$$

where:

Y_{ijk} = The measured value of concentration of a constituent in Replicate j of core i

μ = The mean concentration of the constituent in the tank

C_i = The deviation of concentration in core i from the mean value

S_{ij} = The deviation of concentration in core replicates from the mean value (two replicates were processed on each composite)

E_{ijk} = The analytical (lab) error in the measurements.

All relative standard deviations (RSD) in this appendix are presented as percentages of the mean. The RSD associated with a variance component is the standard deviation of the component divided by μ. The variance components listed in the table are as follows: σ_C is the standard deviation of C_i; σ_S is the standard deviation of S_{ij}; σ_E is the analytical standard deviation.

Table B-1. Tank Concentrations from Composite Samples. (8 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration		ANOVA RSDs			Obs.	
		$\hat{\mu}$	RSD ($\hat{\mu}$)	σ_C	σ_S	σ_E	<DL	#
Anions		(µg/g)						
Chloride	IC:W	1.02e+03	2	NA	NA	4	0	8
Cyanide	CN-:W	1.88e+00	19	0	23	43	1	8
Fluoride	IC:W	1.56e+03	2	3	0	2	0	8
Nitrate	IC:W	8.20e+04	8	11	1	1	0	8
Nitrite	IC:W	4.50e+04	9	13	0	1	0	8
Phosphate	IC:W	2.39e+04	3	4	1	3	0	8
Phosphorus	ICP:A	1.53e+04	10	14	2	2	0	10
Phosphorus	ICP:F	1.59e+04	8	12	0	2	0	8
Phosphorus	ICP:W	7.52e+03	4	6	2	1	0	9
Sulfate	IC:W	1.16e+04	1	NA	NA	3	0	8
Cations		(µg/g)						
Aluminum	ICP:A	8.99e+02	7	10	3	3	0	10
Aluminum	ICP:F	1.36e+03	16	23	3	2	0	8
Aluminum	ICP:W	1.60e+01	NA	NA	NA	NA	8	9
Ammonia	ISE:W	4.58e+01	38	53	10	15	0	8
Antimony	AA (Sb):A	<1.83e+00	NA	NA	NA	NA	8	8
Antimony	ICP:A	1.83e+01	28	0	0	88	6	10
Antimony	ICP:F	<9.84e+01	NA	NA	NA	NA	8	8
Antimony	ICP:W	<1.30e+01	NA	NA	NA	NA	9	9
Arsenic	AA (As):A	<2.91e+00	NA	NA	NA	NA	8	8
Arsenic	ICP:A	<2.79e+01	NA	NA	NA	NA	10	10
Arsenic	ICP:F	<1.57e+02	NA	NA	NA	NA	8	8
Arsenic	ICP:W	<2.08e+01	NA	NA	NA	NA	9	9
Barium	ICP:A	2.82e+01	11	15	3	4	0	10
Barium	ICP:F	4.23e+01	7	0	9	14	0	8
Barium	ICP:W	<2.61e+00	NA	NA	NA	NA	9	9
Beryllium	ICP:A	<1.74e+00	NA	NA	NA	NA	10	10
Beryllium	ICP:F	<9.84e+00	NA	NA	NA	NA	8	8
Beryllium	ICP:W	<1.30e+00	NA	NA	NA	NA	9	9
Bismuth	ICP:A	1.93e+04	2	0	0	6	0	10
Bismuth	ICP:F	2.02e+04	1.	0	0	2	0	8
Bismuth	ICP:W	6.65e+01	7	0	0	20	1	9
Boron	ICP:A	5.14e+01	7	0	11	16	0	10

Table B-1. Tank Concentrations from Composite Samples. (8 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration		ANOVA RSDs			Obs.	
		$\hat{\mu}$	RSD $(\hat{\mu})$	σ_C	σ_S	σ_R	<DL	#
Boron	ICP:F	7.36e+01	25	0	29	59	1	8
Boron	ICP:W	1.53e+01	7	0	0	22	1	9
Cadmium	ICP:A	2.77e+00	15	0	0	49	2	10
Cadmium	ICP:F	2.13e+01	42	57	22	19	1	8
Cadmium	ICP:W	<1.30e+00	NA	NA	NA	NA	9	9
Calcium	ICP:A	6.89e+02	23	33	3	3	0	10
Calcium	ICP:F	8.95e+02	17	23	2	3	0	8
Calcium	ICP:W	1.47e+01	38	35	32	71	3	9
Cerium	ICP:A	3.21e+01	24	0	0	76	4	10
Cerium	ICP:F	<1.57e+02	NA	NA	NA	NA	8	8
Cerium	ICP:W	<2.08e+01	NA	NA	NA	NA	9	9
Chromium	ICP:A	1.11e+03	5	6	3	2	0	10
Chromium	ICP:F	1.15e+03	2	3	0	2	0	8
Chromium	ICP:W	2.67e+02	13	18	0	1	0	9
Cobalt	ICP:A	4.43e+00	21	0	0	65	4	10
Cobalt	ICP:F	2.11e+01	2	2	0	5	2	8
Cobalt	ICP:W	2.66e+00	NA	NA	NA	NA	7	9
Copper	ICP:A	2.01e+02	94	133	5	3	0	10
Copper	ICP:F	2.21e+02	83	118	0	3	0	8
Copper	ICP:W	5.46e+00	69	97	0	25	4	9
Dysprosium	ICP:A	<6.97e+00	NA	NA	NA	NA	10	10
Dysprosium	ICP:F	<3.94e+01	NA	NA	NA	NA	8	8
Dysprosium	ICP:W	<5.21e+00	NA	NA	NA	NA	9	9
Europium	ICP:A	<3.49e+00	NA	NA	NA	NA	10	10
Europium	ICP:F	<1.97e+01	NA	NA	NA	NA	8	8
Europium	ICP:W	<2.61e+00	NA	NA	NA	NA	9	9
Gadolinium	ICP:A	<6.97e+01	NA	NA	NA	NA	10	10
Gadolinium	ICP:F	<3.94e+02	NA	NA	NA	NA	8	8
Gadolinium	ICP:W	<5.21e+01	NA	NA	NA	NA	9	9
Hexavalent Chromium	Calorimetric:W	1.61e+02	6	4	10	3	0	8
Iron	ICP:A	1.64e+04	6	8	3	2	0	10
Iron	ICP:F	1.77e+04	5	7	1	2	0	8
Iron	ICP:W	8.00e+01	5	0	0	15	0	9
Lanthanum	ICP:A	1.13e+01	27	0	0	84	4	10

Table B-1. Tank Concentrations from Composite Samples. (8 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration		ANOVA RSDs			Obs.	
		$\hat{\mu}$	RSD $(\hat{\mu})$	σ_C	σ_S	σ_E	<DL	#
Lanthanum	ICP:F	<5.90e+01	NA	NA	NA	NA	8	8
Lanthanum	ICP:W	<7.82e+00	NA	NA	NA	NA	9	9
Lead	ICP:A	1.57e+03	7	10	2	3	0	10
Lead	ICP:F	1.85e+03	21	2	1	31	0	8
Lead	ICP:IA	1.58e+01	31	0	0	93	6	9
Lithium	ICP:A	<6.97e+00	NA	NA	NA	NA	10	10
Lithium	ICP:F	<3.94e+01	NA	NA	NA	NA	8	8
Lithium	ICP:W	<5.21e+00	NA	NA	NA	NA	9	9
Magnesium	ICP:A	1.95e+02	2	0	3	5	0	10
Magnesium	ICP:F	3.34e+02	8	12	0	2	0	8
Magnesium	ICP:W	2.13e+01	NA	NA	NA	NA	8	9
Manganese	ICP:A	7.89e+01	6	8	3	2	0	10
Manganese	ICP:F	1.11e+02	2	0	0	6	0	8
Manganese	ICP:W	<1.30e+00	NA	NA	NA	NA	9	9
Mercury	CVAA (Hg):A	9.32e+00	50	69	14	24	0	8
Molybdenum	ICP:A	4.17e+01	9	12	5	5	0	10
Molybdenum	ICP:F	5.42e+01	6	8	0	3	0	8
Molybdenum	ICP:A	3.67e+01	9	13	1	2	0	9
Neodymium	ICP:A	2.21e+01	23	20	0	57	1	10
Neodymium	ICP:F	9.42e+01	5	3	0	12	0	8
Neodymium	ICP:W	8.21e+00	30	0	0	89	5	9
Nickel	ICP:A	2.07e+01	7	0	0	22	2	10
Nickel	ICP:W	7.94e+00	NA	NA	NA	NA	8	9
Palladium	ICP:A	5.25e+01	NA	NA	NA	NA	9	10
Palladium	ICP:F	2.99e+02	NA	NA	NA	NA	7	8
Palladium	ICP:W	<3.91e+01	NA	NA	NA	NA	9	9
Potassium	ICP:A	6.74e+02	18	0	0	56	1	10
Potassium	ICP:W	6.19e+02	11	13	0	18	1	9
Rhodium	ICP:A	<3.49e+01	NA	NA	NA	NA	10	10
Rhodium	ICP:F	<1.97e+02	NA	NA	NA	NA	8	8
Rhodium	ICP:W	<2.61e+01	NA	NA	NA	NA	9	9
Ruthenium	ICP:A	<1.74e+01	NA	NA	NA	NA	10	10
Ruthenium	ICP:F	<9.84e+01	NA	NA	NA	NA	8	8
Ruthenium	ICP:W	<1.30e+01	NA	NA	NA	NA	9	9

Table B-1. Tank Concentrations from Composite Samples. (8 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration		ANOVA RSDs			Obs.	
		$\hat{\mu}$	RSD $(\hat{\mu})$	σ_C	σ_g	σ_E	<DL	#
Selenium	AA (Se):A	<1.46e+01	NA	NA	NA	NA	8	8
Selenium	ICP:A	3.23e+01	22	0	0	69	4	10
Selenium	ICP:F	<1.48e+02	NA	NA	NA	NA	8	8
Selenium	ICP:W	<1.95e+01	NA	NA	NA	NA	9	9
Selenium	Liquid Scintillation:F	7.35e-05	32	44	8	10	0	8
Silicon	ICP:A	4.91e+02	21	30	0	5	0	10
Silicon	ICP:F	1.04e+04	8	12	0	1	0	8
Silicon	ICP:W	6.53e+02	3	0	4	4	0	9
Silver	ICP:A	5.95e+00	26	30	0	47	2	10
Silver	ICP:F	9.74e+01	32	45	11	7	0	8
Silver	ICP:W	2.66e+00	NA	NA	NA	NA	8	9
Sodium	ICP:A	8.79e+04	2	2	3	2	0	10
Sodium	ICP:F	9.57e+04	2	2	2	2	0	8
Sodium	ICP:W	8.05e+04	0	0	1	1	0	9
Strontium	ICP:A	2.18e+02	2	0	3	2	0	10
Strontium	ICP:F	2.21e+02	2	2	0	2	0	8
Strontium	ICP:W	1.39e+00	29	0	0	86	3	9
Tellurium	ICP:A	3.60e+01	28	0	0	90	6	10
Tellurium	ICP:F	<1.97e+02	NA	NA	NA	NA	8	8
Tellurium	ICP:W	<2.61e+01	NA	NA	NA	NA	9	9
Thallium	ICP:A	<1.74e+02	NA	NA	NA	NA	10	10
Thallium	ICP:F	<9.84e+02	NA	NA	NA	NA	8	8
Thallium	ICP:W	<1.30e+02	NA	NA	NA	NA	9	9
Tin	ICP:A	<2.79e+02	NA	NA	NA	NA	10	10
Tin	ICP:F	<1.57e+03	NA	NA	NA	NA	8	8
Tin	ICP:W	<2.08e+02	NA	NA	NA	NA	9	9
Titanium	ICP:A	7.90e+00	14	19	0	10	0	10
Titanium	ICP:F	2.86e+01	4	5	1	5	0	8
Titanium	ICP:W	1.30e+00	NA	NA	NA	NA	9	9
Tungsten	ICP:A	<2.79e+01	NA	NA	NA	NA	10	10
Tungsten	ICP:F	<1.57e+02	NA	NA	NA	NA	8	8
Tungsten	ICP:W	<2.08e+01	NA	NA	NA	NA	9	9
Uranium	ICP:A	4.13e+02	23	0	0	73	4	10
Uranium	ICP:F	<1.97e+03	NA	NA	NA	NA	8	8

Table B-1. Tank Concentrations from Composite Samples. (8 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration		ANOVA RSDs			Obs.	
		$\hat{\mu}$	RSD $(\hat{\mu})$	σ_C	σ_S	σ_R	<DL	#
Uranium	ICP:W	2.73e+02	30	0	0	89.	4	9
Uranium	Laser Fluorimetry:F	1.97e+02	4	6	1	2	0	8
Vanadium	ICP:A	3.93e+00	25	0	0	79	4	10
Vanadium	ICP:F	<1.97e+01	NA	NA	NA	NA	8	8
Vanadium	ICP:W	<2.61e+00	NA	NA	NA	NA	9	9
Yttrium	ICP:A	3.93e+00	25	0	0	79	5	10
Yttrium	ICP:F	<1.97e+01	NA	NA	NA	NA	8	8
Yttrium	ICP:W	<2.61e+00	NA	NA	NA	NA	9	9
Zinc	ICP:A	1.11e+02	50	71	3	3	0	10
Zinc	ICP:F	1.73e+02	23	33	3	5	0	8
Zinc	ICP:W	<5.21e+00	NA	NA	NA	NA	9	9
Zirconium	ICP:A	1.44e+01	29	41	0	10	0	10
Zirconium	ICP:F	2.05e+01	2	0	0	6	4	8
Zirconium	ICP:W	<2.61e+00	NA	NA	NA	NA	9	9
Organics		(µg/g)						
1,2,4-Trichlorobenzene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
1,2-Dichlorobenzene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
1,3-Dichlorobenzene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
1,4-Dichlorobenzene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2,4,5-Trichlorophenol	SVOA	<4.81e+01	NA	NA	NA	NA	8	8
2,4,6Trichlorophenol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2,4-Dichlorophenol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2,4-Dimethylphenol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2,4-Dinitrophenol	SVOA	<4.81e+01	NA	NA	NA	NA	8	8
2,4-Dinitrotoluene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2,6-Dinitrotoluene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2-Chloronaphthalene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2-Chlorophenol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2-Methylnaphthalene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2-Methylphenol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
2-Nitroaniline	SVOA	<4.81e+01	NA	NA	NA	NA	8	8
2-Nitrophenol	SVOA	<9.61e+00	NA	NA	NA	NA1	8	8
3,3-Dichlorobenzidine	SVOA	<1.94e+01	NA	NA	NA	NA	8·	8
3-Nitroaniline	SVOA	<4.81e+01	NA	NA	NA	NA	8	8

Table B-1. Tank Concentrations from Composite Samples. (8 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration		ANOVA RSDs			Obs.	
		$\hat{\mu}$	RSD ($\hat{\mu}$)	σ_C	σ_S	σ_R	<DL	#
4,6-Dinitro-o-cresol	SVOA	<4.81e+01	NA	NA	NA	NA	8	8
4-Bromophenylphenyl ether	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
4-Chloro-3-methylphenol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
4-Chloroaniline	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
4-Chlorophenylphenyl ether	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
4-Methylphenol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
4-Nitroaniline	SVOA	<4.81e+01	NA	NA	NA	NA	8	8
4-Nitrophenol	SVOA	<4.81e+01	NA	NA	NA	NA	8	8
Acenaphthene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Acenaphthylene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Anthracene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Benzo(a)anthracene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Benzo(a)pyrene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Benzo(b)fluoranthene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Benzo(ghi)perylene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Benzo(k)fluoranthene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Benzoic acid	SVOA	<4.81e+01	NA	NA	NA	NA	8	8
Benzyl alcohol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Bis(2-Chloroethoxy)methane	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Bis(2-chloroethyl) ether	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Bis(2-chloroisopropyl)	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Bis(2-ethylhexyl) phthalate	SVOA	2.73e+00	8	9	0	15	0	8
Butylbenzylphthalate	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Chrysene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Decane	SVOA	1.68e+01	16	NA	NA	24	0	4
Di-n-butylphthalate	SVOA	8.44e+00	NA	NA	NA	NA	7	8
Di-n-octylphthalate	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Dibenz[a,h]anthracene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Dibenzofuran	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Diethylphthalate	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Dimethyl phthalate	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Dioctyl adipate	SVOA	1.20e+01	17	22	0	20	0	8
Dodecane	SVOA	7.96e+02	68	95	15	20	0	8
Extractable total organic halides	Ext Org Halides	<1.00e+01	NA	NA	NA	NA	8	8

Table B-1. Tank Concentrations from Composite Samples. (8 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration		ANOVA RSDs			Obs.	
		$\hat{\mu}$	RSD $(\hat{\mu})$	σ_C	σ_S	σ_E	<DL	#
Fluoranthene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Fluorene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Hexachlorobenzene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Hexachlorobutadiene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Hexachlorocyclopentadiene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Hexachloroethane	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Indeno(1,2,3-cd)pyrene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Isophorone	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
N-Nitroso-di-n-dipropylamine	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
N-Nitrosodiphenylamine	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Naphthalene	SVOA	<,9.61e+00	NA	NA	NA	NA	8	8
Nitrobenzene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Pentachlorophenol	SVOA	<4.81e+01	NA	NA	NA	NA	8	8
Pentadecane	SVOA	5.50e+01	60	84	13	20	0	8
Phenanthrene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Phenol	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Pyrene	SVOA	<9.61e+00	NA	NA	NA	NA	8	8
Tetradecane	SVOA	1.14e+03	49	69	1	13	0	8
Total carbon	Persulf. Oxidation:D	4.80e+03	2	0	2	3	0	8
Total carbon	Persulf. Oxidation:W	5.34e+03	7	10	0	3	0	8
Total inorganic carbon	Persulf. Oxidation:D	3.73e+03	11	15	0	3	0	8
Total inorganic carbon	Persulf. Oxidation:W	4.46e+03	11	14	0	9	0	8
Total organic carbon	Persulf. Oxidation:D	1.07e+03	37	51	14	6	0	8
Total organic carbon	Persulf. Oxidation:W	8.75e+02	12	0	0	35	0	8
Tributyl phosphate	SVOA	2.20e+01	14	NA	NA	23	0	4
Tridecane	SVOA	1.73e+03	54	75	22	5	0	8
Undecane	SVOA	3.55e+01	15	NA	NA	23	0	4
Physical Properties								
pH Measurement	pH:W	8.87e+00	1	2	NA	0	0	4
Weight percent solids	Percent Solid:D	3.69e+01	2	2	0	0	0	8
Radionuclides		(µCi/g)						
Americium-241	Alpha Radchem:F	6.94e-02	20	27	7	5	0	8
Americium-241	GEA:F	8.46e-02	25	22	36	19	0	8
Carbon-14	Liquid Scintillation:W	8.28e-03	41	0	0	115	6	8

Table B-1. Tank Concentrations from Composite Samples. (8 sheets)

Analyte	Analytical Method: Sample Preparation	Mean Concentration		ANOVA RSDs			Obs.	
		$\hat{\mu}$	RSD ($\hat{\mu}$)	σ_C	σ_S	σ_R	<DL	#
Carbon-14	Liquid Scintillation:W	1.60e-03	36	0	28	94	0	8
Cesium-137	GEA:F	1.58e+02	9	12	0	6	0	8
Cobalt-60	GEA:F	<3.87e-03	NA	NA	NA	NA	8	8
Curium-242	Alpha Radchem:F	9.16e-05	29	40	0	12	0	6
Curium-243/244	Alpha Radchem:F	4.70e-04	57	0	83	111	0	8
Europium-154	GEA:F	1.70e-01	26	36	7	9	0	8
Europium-155	GEA:F	2.00e-01	30	42	6	11	0	8
Gross alpha	Alpha Radchem:F	1.76e-01	6	7	4	6	0	8
Gross beta	Beta Radchem:E	6.28e+02	15	22	1	2	0	8
Neptunium-237	Alpha Radchem:F	7.14e-05	22	22	29	19	0	8
Plutonium-238	Alpha Radchem:F	3.05e-03	10	6	17	12	0	8
Plutonium-239/240	Alpha Radchem:F	9.73e-02	5	0	9	9	0	8
Strontium-90	Beta Radchem:F	2.48e+02	22	31	6	9	0	8
Technetium-99	Beta Radchem:F	1.14e-01	10	14	2	3	0	8
Thorium-232	ICP:A	<2.79e+02	NA	NA	N A	NA	10	10
Thorium-232	ICP:F	<1.57e+03	NA	NA	NA	NA	8	8
Thorium-232	ICP:W	<2.08e+02	NA	NA	NA	NA	9	9
Total alpha	Alpha Radchem:F	1.00e-01	5	0	8	9	0	8
Tritium	Liquid Scintillation:W	2.75e-03	15	19	0	16	0	8
		(%)						
Uranium-234	Mass Spectrometry:F	5.27e-03	7	5	0	17	0	8
Uranium-235	Mass Spectrometry:F	6.62e-01	0	0	0	0	0	8
Uranium-236	Mass Spectrometry:F	9.35e-03	5	0	0	14	0	8
Uranium-238	Mass Spectrometry:F	9.93e+01	0	0	0	0	0	8

*Total alpha emitted by ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu.

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APPENDIX C

RAW DATA SET SUMMARY

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APPENDIX C

RAW DATA SET SUMMARY

This appendix describes the format of the B-111 data set used to produce the results discussed in this report. The data set contains chemical measurements made by the 325-A Laboratory on B-111 core samples. The data were originally downloaded from the Tank Characterization Database (TCD). The following changes were made to the data set in preparation for the various statistical analyses:

1. The potassium hydroxide fusion inductively coupled plasma (ICP) analyses for nickel and potassium were removed from the data set.

2. Only 17 of the original 40 TCD fields remain in the data set.

3. Any sample result that was below the detection limit was replaced with the detection limit value, if it was available.

4. All of the toxic characteristic leach procedure results by the acid digestion ICP analysis method were removed, to avoid confusion with the standard acid digestion ICP analyses.

5. The organics results were converted from parts per billion to parts per million.

An electronic ASCII copy of the B-111 data set is available upon request. This data set does not include any of the quality, assurance data (i.e., matrix spikes and method blanks). The B-111 data set is 5,109 records in length. Table C-1 describes the contents of each field. DOE [1994] contains more information on the format of the data in the TCD.

Table C-2 contains an example of three records from a dataset similar to the B-111 dataset.

REFERENCES

DOE, 1994, *Hanford Environmental Information System (HEIS), Tank Characterization Data (TCD) Subject Area*, DOE/RL-93-24-8, Volume 8, U.S. Department of Energy, Richland Operations Office, Richland, Washington.

Table C-1. Description of Tank 241-B-111 Data Set Fields.

Field	Description
1	Core Number
2	Segment or Composite Number
3	Analytical Method Name
4	Phase of the Waste Sample (i.e, Solid or Liquid)
5	Sample Location (TOP and BOTTOM are homogenization samples and TOTAL is the standard sample)
6	Sample ID Number (Assigned by the 325-A Laboratory)
7	Dilution Factor
8	Sample Batch Number
9	Table and Page Number in the Validation Report that contain the sample results
10	Constituent name
11	Measured Sample Result
12	Result Type (e.g., Primary Result, Duplicate Result)
13	Result Units
14	Detection Limit
15	Detection Limit Units
16	Data Quality Flags assigned by Hanford Analytical Services
17	Field indicating if a result is above the detection limit (T = above DL, F = below DL)

Table C-2. Example of Three Records from a Raw Data Set.

Field 1	Field 2	Field 3	Field 4	Field 5
Field 6	Field 7	Field 8	Field 9	Field 10
Field 11	Field 12	Field 13	Field 14	Field 15
Field 16	Field 17			
core26	3	Extraction Organic (VOA)	S	TOTAL
BLANK	1.0		PG 145	Tetrachloroethane
3.800000e+06	PRIMARY_RESULT	UG/G	NA	
UDR	F			
core26	3	Acid Digestion ICP	S	TOP
9203238A	10.0	21	Pg 67, Table 2-2e	Tellurium
2.087700e+02	DUPLICATE_RESULT	UG/G	208.77000	UG/G
U	F			
core27	Comp1	Fusion ICP	S	BOTTOM
9210669H1B	2.0	49	Pg 353, Table 2-1b	Tellurium
4.293200e+02	DUPLICATE_RESULT	UG/G	429.32000	UG/G
U	F			

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W. C. Miller	R1-30	X			
C. T. Narquis	T6-16	X			
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